

TOPICS OF THE MONTH

Two symposia—many impressions

TWO remarkable chemical engineering symposia took place in Europe recently. The first, held in Amsterdam, was on chemical reaction engineering; the second, in Brighton, was on distillation. (Full reports of these symposia are included in this issue.) The cynic may argue that such gatherings serve but to provide a happy social hunting-ground, since pre-prints of the papers are in every case available prior to the meetings. Would it not be simpler, therefore, to study these papers in the privacy of one's own (laboratory) arm-chair?

It must be borne in mind that there are two sides to every international congress; the excellence is often judged as much by the quality of papers presented as by the social contacts made. Unhappily, the political bisection of the world has made scientific intercommunication with many countries quite difficult. The very notion of an iron curtain in science is anathema to the spirit of pure research, and international congresses are one proof that such a curtain is being dissolved. Evidence of this was readily available both at Amsterdam and Brighton, where many chemical engineers could for the first time gauge the international importance of their subject. Language barriers were easily surmounted and a free exchange of ideas was made as much within the conference room as across the lunch tables.

One overriding impression created at these symposia was the high standard of fundamental research work carried out at universities and research associations. This work is of such a specialised and profound nature that industry could never hope to equal it. This should in no way detract from the excellent applied research undertaken by the large industries; however, most industrial engineers must rely upon fundamental published research from the universities. One aim of such gatherings is, therefore, to diffuse available knowledge, which can be slowly assimilated and applied by industry.

What are the inherent drawbacks to such canalised research? The plant engineer in industry often claims that fundamental work at university is only carried out on simple near-ideal systems. If vapour-liquid equilibria are studied, for example, binary hydrocarbon systems are mostly selected and investigated. How many multi-component complex systems are ever rigorously investigated? In industry, however, the engineer is often confronted with multi-component distillation problems and will find it next to impossible to trace any previous work on such systems. As a result he must make his own assumptions based on available knowledge of simple systems. Very often he does not possess sufficient training to make the correct assumptions and consequently mistakes are liable to occur. More often than not he will just neglect any previous knowledge and treat his problem empirically.

It can be argued by research workers, on the other hand, that before the more complicated systems can be tackled simplified systems must first be understood; in other words, we must learn to walk before we can run. Nobody denies the soundness of this argument, yet what is often necessary and not forthcoming is the guiding hand extended from university to industry. A reversible diffusion of ideas and information must surely result in a better grasp of existing problems throughout the profession. If the aftermath of Amsterdam and Brighton can bring this about, these symposia will indeed be regarded as milestones in the history of chemical engineering.

Deuterium-hydrogen separation

ALL naturally occurring hydrogen compounds have approximately the same deuterium to hydrogen ratio as hydrogen itself, 1 part in 6,000 to 7,000. The increasing trend towards heavy water reactors has stressed the importance of economically separating deuterium from hydrogen. It has been shown that if this hydrogen were produced solely as a deuterium source its cost would be exceedingly high. To reduce this cost heavy water plants are often made parasites to plants producing hydrogen for other purposes, e.g. ammonia synthesis. The main disadvantages of this are that deuterium production is tied to the location of ammonia plants and their capacity does not represent the optimum production capacity of a heavy water unit.

Water, being the most abundant source of hydrogen, is therefore always considered as a source of deuterium, and this can be obtained by water distillation, exchange and electrolysis. A heavy water plant requiring a small capital investment has been suggested by T. M. Flynn in the March 1960 issue of *Chemical Engineering Progress*. Such a plant has three principal components:

- (1) A source of river, lake or sea-water.
- (2) A hydrogen-water exchange reactor.
- (3) A hydrogen-deuterium stripper.

The exchange reactor differs from other more widely used exchange reactors in two important aspects: firstly, it is intended to enrich the hydrogen stream rather than the water and, secondly, the degree of enrichment is only up to the natural abundance. It has been shown that when two pairs of isotopic compounds, H_2O , HDO and H_2 , HD are moved counter-current to one another in different phases, separation of deuterium from hydrogen is attained. The hydrogen-deuterium exchange can be carried out either in a simple exchanger for chemical conversion of one deuterium-rich compound to another or in a dual-temperature exchanger. Most important in this plant is the distillation process, in which deuterium is separated (stripped) from hydrogen. Very little work has been carried out on this problem and only recently the

National Bureau of Standards' Cryogenic Engineering Laboratory conducted a research programme to evaluate some design parameters for hydrogen distillation. In order to overcome thermal leakage from the atmosphere into the column it was insulated with two concentric radiation shields at 76°K. and 20°K., all enclosed in a high vacuum space. It was found that the H₂-HD separation by fractional distillation is not a difficult one and that the number of equilibrium stages required is not large. The pilot plant designed at the cryogenic laboratory was capable of increasing the concentration of HD in H₂ from 0.03% to 3% and has a capacity equivalent of 48 lb. heavy water in an 8,000-hr. year. Overall plate efficiencies in this range of vapour velocities were found to vary from 45% to 55%.

The production of deuterium is only one of the numerous applications which require a specialised knowledge of cryogenics engineering. Our special feature on cryogenics draws attention to many interesting new developments which have been achieved in the realm of low-temperature physics and engineering.

Industrial research expenditure in 1958

BRITISH manufacturing industry spent about £300 million on research and development in 1958, compared with about £190 million in 1955. This represents 4.2% of manufacturing industry's contribution to national production, compared with 3.1% in 1955. 95% of the £300 million was spent in industry's own establishments and about 5% on payment to outside bodies, such as co-operative research associations, universities and other public and private research institutions.

These estimates are contained in a D.S.I.R. survey on 'Industrial Research and Development Expenditure 1958', which was recently published. The main expenditure on research and development for the year under review was in aircraft (£100 million), electrical engineering (£64 million) and chemicals (£43 million). These estimates of expenditure are based on the estimates of the distribution of qualified scientists and engineers engaged on research and development provided by a survey carried out in January 1959 by the Ministry of Labour and National Service for the Advisory Council on Scientific Policy. D.S.I.R. carried out a special enquiry designed to discover how the cost of employing a qualified worker on research and development had changed since 1955. The results of this enquiry, described in the report, show that this cost varies greatly from one industry to another and is related to the amount of development expenditure and the use made of supporting (not 'fully qualified') staff. The average cost of a qualified worker for the chemical industry was £6,600. In the case of the aircraft industry the figure was nearly £31,000 and the average for the manufacturing industry other than aircraft was just under £8,000.

The report shows that there were 20,500 personnel employed in research and development in the chemical industries' own research establishments; 31% of these were qualified scientists and engineers.

Royal Society

LIKE several other notable British institutions, the Royal Society is unique throughout the world. To paraphrase Francis Bacon:

Magna ista scientiarum mater.

Surely the Royal Society is mother of all scientific institutions everywhere! This year we are celebrating the 300th anniversary of its foundation and it is well to recall some of its early history. In its royal charter, granted by Charles II in 1662, the Society's purpose is stated as being the promotion of 'natural knowledge'. This the Society has endeavoured to fulfil in various ways, chiefly by publications of new scientific work and subsequent discussions. The *Philosophical Transactions of the Royal Society* are considered by many as the finest scientific periodical in the world, although due to the diversity of subjects included therein it is often difficult for the specialist to extract those papers which bear on his subject.

It is interesting to note that Isaac Newton was elected a Fellow in 1671 at the age of 28, after his presentation to the Society of a reflecting telescope made with his own hands. As a result of the interest aroused by the telescope he sent the Society his first paper, which was published in the *Philosophical Transactions* in 1672. In 1703 Newton became president of the Society, an office which he held until his death in 1727. Amongst other illustrious past presidents of the Society must be included Sir Christopher Wren, Samuel Pepys, Sir Joseph Banks, Sir Humphrey Davy, Sir Joseph Hooker, Lord Lister and Lord Rutherford.

Nowadays the Society takes part in the work of U.N.E.S.C.O. in the field of the natural sciences through a special committee whose chairman is the foreign secretary of the Society. The Society is also responsible for the ultimate direction of the scientific work of the National Physical Laboratory and plays a part in the management of the Royal Greenwich Observatory and the Isaac Newton Observatory.

It must be stressed that the Society is completely independent of direct Government support, as indeed is proved by the stiff membership contributions which Fellows have to pay annually. Throughout the past three centuries the principles of the Society have remained the same: to promote the reliance on experiment and observation rather than on authority and to further the friendly co-operation of learned men without boundaries of race or creed.

Future of zinc oxide

THE production of zinc oxide is a useful geiger counter to detect activity in many branches of the chemical industry. In a recent lecture at the annual meeting of the American Zinc Institute, R. H. Crossley analysed the trends in the use of zinc oxide. He pointed out that during the past decade the rubber and paint industries have consumed about 80% of the total zinc oxide produced. However, a significant change in the proportionate use of zinc oxide by these two industries can be detected. Consumption by the rubber

industry has shown a fairly steady increase and a consumption of 100,000 tons p.a. is predicted by 1965, as compared with 88,000 tons in 1950. In contrast, consumption by the paint industry has shown a steady decline from 104,000 tons in 1950 to 60,000 tons in 1959. It is estimated that the 1965 figures will be about 65,000 tons. All other uses of zinc oxide remain fairly constant at about 40,000 tons p.a. and no great change in this category is expected, since no important new uses have been developed.

It is apparent, therefore, that the principal reason for the failure of zinc oxide to keep pace with industrial production as a whole has been its decline in use by the paint industry. The rubber industry also has problems which affect zinc oxide. As an example, rubber products are meeting increasing competition from products of the non-rigid plastics industry.

It was stressed that the most important single reason for the decline in the use of zinc oxide by the rubber and paint industries has been its cost. Products of these industries are sold on a volume and not weight basis. Hence the volume cost of ingredients is important. Zinc oxide, having a specific gravity of 5.6, is often the most expensive ingredient in a rubber or paint formula. To counter this defect zinc oxide producers have tried to improve their products by surface treatment, to improve dispersion, and pelletising to reduce handling and storing costs. The outlook for zinc oxide thus is far from bright. It is now up to zinc oxide producers to actively persuade a reluctant industry to purchase more than the minimum of this chemical.

Metals for high temperatures

HOW can metals be developed to withstand the ever-higher temperatures of many modern chemical and nuclear reactions? For purely metallic materials the melting point of tungsten at 6,170°F. can be taken as the upper limit. If refractory hard metal compounds are included among the metallic materials the limit is slightly raised. Tantalum carbide, for example, melts only at about 6,800°F. and hafnium carbide at about 7,000°F. Below this upper limit there is a broad range of metals with melting points extending down to almost room temperature.

Many attempts have been made to relate the melting point of metals to tensile strength. Although no quantitative relationship has been established, it is known that at least qualitatively the melting point and tensile strength of metals are related. As a result of this, fundamental relationships have become the starting points in the development of new high-temperature metals and alloys. Allen and Carrington in the *Journal of the Institute of Metals* (1954, 82/2) have shown that there is a periodicity in creep strength. Thus chromium, molybdenum and tungsten, the Group VI metals, show maximum values as the fourth, fifth and six periods of the periodic table are traversed. Columbium and tantalum, which together with vanadium make up the Group V metals, are also outstandingly strong. The six metallic elements in Groups V and VI, or combinations of them, offer the best hope of meeting the

very high-strength, high-temperature demands of metallic structures. Other elements at high temperatures are either too weak or too scarce.

Extensive development work is being done on alloys of the refractory materials. It has been established, for example, that small amounts of alloying elements strengthen molybdenum—a $\frac{1}{2}\%$ addition of titanium increases the 100-hr. rupture stress at 1,800°F. from 18,000 to 45,000 p.s.i. Recently important improvements in the strength of tungsten have been observed after the addition of a few per cent. of ThO_2 . Thus a 2% dispersion of ThO_2 raises the tensile strength at 2,500°F. from 22,000 to 42,000 p.s.i.

Refractory metals and their alloys are only used nowadays when absolutely necessary. Metallic materials are preferred as a rule, because they are normally cheaper and easier to fabricate and sometimes even offer better strength-weight ratios as well as oxidation resistance. The other metallic materials can be divided into the super-alloys, which have nickel or cobalt bases, and the ferritic, martensitic and austenitic steels, which have ferrous bases.

Japanese chemical statistics

RECENTLY published figures show that in common with the rest of the western world the Japanese economy expanded quite markedly during the period April-September 1959. However, one characteristic feature of this prosperity was a wide discrepancy in the activities of many industries.

The output of ammonia-derivative fertilisers, for example, totalled 2 million tons in terms of ammonia sulphate, a decline of 8% as compared with the previous period. This decline was a result of the curtailment caused by pressure from huge accumulated stocks. This industry seems to have been confronted with a difficult situation caused by stagnation in the home market, coupled with intensified international competition. The total production of superphosphates, amounting to 920,000 tons, represented a decrease of 3% as compared with the previous period, due to the lower seasonal demand. Nevertheless, the superphosphate industry did present some extent of stability, although at a lower level of profits.

The output of caustic soda, 370,000 tons, showed a marked increase, amounting to 12% above the previous period. This was attributed to the increasing demand from major consumers, notably the pulp, paper and textile industries. The demand for chlorine as by-product was considerably increased, notably from vinyl chloride for PVC.

With the petrochemical industry gradually entering into the Japanese economy, expansion in this field was most significant in the production of high-pressure polyethylene. Production of vinyl chloride was 95,000 tons during this period, and this was 56% above the previous term. The output of fuel oil was increasing rapidly to 9,160 million litres, 16% above the previous period. The oil-refining industry was, of course, favoured by the world-wide excess in crude oil and decline in freight rates.

American uranium production

THE U.S. Atomic Energy Commission recently announced figures on domestic uranium production for the last six months of 1959. The measured and inferred ore reserves, including only material metallurgically amenable to treatment, was estimated to total 86 million tons on December 31, 1959. The breakdown per state is as follows:

State	Thousand tons	U_3O_8 %
New Mexico	55,700	0.26
Wyoming	15,800	0.34
Utah	5,300	0.33
Colorado	4,500	0.30
Arizona	1,200	0.35
Washington, Oregon and Nevada	1,900	0.21
North and South Dakota	600	0.27
Others (Texas, California, Montana, Idaho and Alaska)	1,100	0.24
Total reserves (rounded)	86,100	0.28

In addition, ore stockpiles totalled 1,449,069 dry tons as of December 31, 1959. Ore receipts at all private plants and government purchase depots in July to December 1959 totalled 3,614,000 dry short tons, and ore fed to processes totalled 3,623,000 tons, with an average grade of 0.24% U_3O_8 . Uranium concentrates received at the Grand Junction depot totalled 7,899 tons of U_3O_8 in the six months period.

There are 25 privately owned uranium processing mills operating in the western United States at the present time. The combined rated daily capacity of these mills during that period was 22,100 tons of ore, and their total estimated capital investment was about \$142 million.

The average price paid by the Government for the concentrates was \$8.79/lb., or approximately \$139 million. An additional 90 tons were produced as a by-product in the chemical processing of phosphate rock in Florida and Illinois, and from treatment of Idaho euxenite at the Mallinckrodt Chemical Co.

It is interesting to note that the uranium content of American ore (0.28%) is far higher than Canadian ore (0.086%) or South African ore (0.0285%). As an approximation it would be correct to assume that the total world reserves for uranium element at present are about 2 million metric tons.

Co-polymers in the Soviet Union

SOVIET research in polymer technology was described in London recently when Dr. N. S. Akutin, director of the Russian Institute of Plastics, read a paper on graft and block co-polymerisation to the London section of the Plastics Institute. Dr. Akutin described new techniques in this field, such as polymerisation by means of ultrasonic waves and by electric spark discharge, which are rapidly becoming of industrial importance in the Soviet Union. A better-known polymerisation technique used nowadays is one in which polymerisation is initiated by formation of free radicals or ions by grinding or abrading a polymer with an inorganic powder. The powder is ruptured in a homogenising mill to form free radicals or ions that

subsequently initiate polymerisation to graft on to the homopolymer.

Work along similar lines was carried out by Watson at the British Rubber Producers' Research Association in 1956, whereby co-polymers of natural rubber with styrene and methyl methacrylate were obtained by shearing in an inert atmosphere. However, Watson's process has not been applied on a large scale in this country, whereas the Russians claim that they are at the moment producing such graft co-polymers as phenol formaldehyde-coke and phenol formaldehyde-nitrile rubber on an industrial scale. These are mainly used for their high chemical and thermal resistance in chemical plant fabrication.

Hitherto, most western polymer scientists and technologists have only been acquainted with Kargin's work in the Soviet Union. It would be a mistake to underestimate the vast amount of research carried out in all spheres of polymer development in Soviet Russia, and western workers should keep abreast of this work.

Oasis in Turkey

DECADES of endeavour by international agencies have been required to convince most people that English is today the lingua franca of science and technology. The 20th century has seen a greater upsurge of chauvinism than any previous century in human civilisation and coupled with this there has always been the insistence by newly-established states to develop their native tongues to the utmost. Whereas this is of undoubted interest to philologists, it has become most disruptive to the ready interchange of technical information and has forced most technical people to acquaint themselves with a working knowledge of three or four languages.

We therefore welcome the establishment of a new technical university in Turkey, where instruction will be given in English only. This university has certain unusual features. It is independent of all government control; its purpose is to serve as a regional university for the whole Middle East. Although at present it is governed by a Turkish board of trustees, it will have strong international affiliations. The president of the university is Dr. E. S. Burdell, formerly president of the Cooper Union for the Advancement of Science and Art in New York. Prof. H. Davies, of Queen Mary College, University of London, has been acting as adviser to the Foreign Office on the university and has been associated with this venture from its inception. There is already a substantial nucleus of British staff, amongst them Prof. G. Bibby, appointed as professor of electrical engineering, and Mr. E. H. K. Dibden, former secretary of the Cavendish Laboratory, who is the new registrar.

At present the number of students totals 550, most of whom are Turkish. Big problems still face the university, especially as regards finance, administration and staff. This may, however, become a precedent to the internationalisation of technology, since it is hoped that students all over the Middle East will flock to this Mecca of learning.

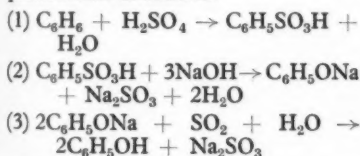
Solvent Extraction of Phenol from Sulphite Liquors—Plant Design

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One method of producing synthetic phenol is by directly fusing benzene sulphonic acid with caustic soda. The resulting sodium phenate can be converted to phenol by several methods. One of these, which is considered here, is springing with sulphur dioxide gas. Since the process economics are dependent on the highest recovery of phenol and sodium sulphite, the author has described a special plant designed for extracting phenol from sulphite liquors followed by dephenolation of the extract with caustic soda.

A RECENT modification to the classical sulphonation process for the manufacture of synthetic phenol involves the direct fusion of 75% W/W NaOH with benzene sulphonic acid ($C_6H_5SO_3H$). This process requires a smaller capital investment for the same production and in addition provides appreciable savings in labour and fuel requirements over the old process, thereby rendering this a more serious competitor to the Raschig Durez process than as reported by Messing and Keary.¹

The reactions involved in the preparation are as follows:



The process economics are still dependent, however, on the highest possible recovery of phenol and sodium sulphite, the latter in a good commercial grade of purity. Since it is only at the stage presented by equation (3) that phenol is naturally separated, it is essential that at this and successive purification stages maximum recovery of phenol must be achieved.

The concentrated solution of sodium phenate is prepared in the fusion pots according to equation (2) and the sodium phenate content of $30\% \pm 5\%$ V/V is controlled in the fusion process to produce this solution which has the lowest viscosity (1.4 cp.) at the working temperature of 70°C. consistent

with a minimum solubility of sodium sulphite in the phenate solution at this temperature (0.46 g./100 g.).

Addition of water

Addition of water then takes place at the springing plant prior to splitting with SO_2 gas, to reduce the concentration to 20% V/V sodium phenate, since this is the highest operating concentration for the removal of the by-product sodium sulphite liquors.

These liquors contain some 18% W/W sodium sulphite at a temperature of only a degree or so above ambient, and is the highest concentration possible for all-year-round pumping through unlagged pipes.

In practice, some sodium sulphite is carried over with the phenate in solution and suspension, and must be taken into account during the splitting or springing operation for the release of the (crude) synthetic phenol. This springing operation can be carried out by any of the following methods:

- (1) Addition of dilute sulphuric acid to give a concentrated solution of sodium sulphate and crude phenol.
- (2) Carbonating with CO_2 to give sodium carbonate solution with crude phenol; and
- (3) Springing with dry 7% SO_2 gas from a contact acid plant to produce a concentrated sodium sulphite solution with crude phenol.

All these methods are dependent for operational efficiency on the production of a final mixture which will

rapidly settle into two immiscible components. To ensure that as far as possible the components are immiscible it is essential to work with the maximum concentration in the aqueous solution consistent with the lowest viscosity of the solution at the plant working temperature.

Consideration of processes

(1) **Weak acid springing:** This is the method which has been most generally favoured both on the Continent, in the U.K. and in the U.S.A. It does, however, suffer from the serious disadvantage that sodium sulphate is not very soluble and possesses an inverted solubility curve at higher temperature. The sodium sulphate solution produced is commercially of little value and is not worth concentration to sell as crude sulphate crystal. At the working concentration the crude phenol solubility is quite high (2.5 to 3% W/W) and this fact, together with the presence of suspended phenol, renders this a most difficult liquor for effluent disposal, to such a degree that it must pass through either a solvent extraction or a steam stripping column before running to drain.

(2) **Carbonating with CO_2 :** This method is the one favoured in Germany for the splitting of phenate solution. For efficient operation it requires either:

- (a) The plant to operate under pressures of up to 5 atm.; or

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(b) A supply of CO_2 at a concentration exceeding 90%.

The latter can be ensured in an adequate state of purity by the newer CO_2 plants which absorb CO_2 from flue gas in monoethanolamine/potassium carbonate solutions and regenerate the CO_2 by steam stripping. The main advantage of the process lies in the fact that the sodium carbonate liquor produced, when stripped of its phenol content by solvent extraction, can be efficiently causticised to recover the soda content for further fusion operations.

(3) **Springing with SO_2 :** This is the favoured method for plants producing phenol by direct fusion without the neutralisation step, since it ensures that the sole by-product consists of sodium sulphite. If this sulphite is stripped of its phenol content by solvent extraction it can form a valuable by-product and is sold as commercially pure anhydrous sodium sulphite crystals. In the neutralisation process for synthetic phenol manufacture the wet SO_2 from the neutralisation of sodium sulphite with benzenesulphonic acid ($\text{C}_6\text{H}_5\text{SO}_3\text{H}$) is used for springing phenate solution in this same manner. The resulting solution of sodium sulphite is recycled to the neutralisation stage.

In this case it is not so essential to strip the liquors of their phenol content, though this is still advisable if maximum phenol recovery is required. In one plant where the direct fusion process for synthetic phenol manufacture is operated, part of the sodium sulphite liquor is used to produce caustic soda by causticising with lime. In the preparation of this caustic soda the remaining part of the uncausticised sodium sulphite in solution is concentrated and salted out in crystallising evaporators.

It is essential that this solution be stripped of its phenol content since otherwise the sodium phenate resulting from the causticising operation acts as an inhibitor in the formation and growth of the sodium sulphite crystals and renders these difficult to filter off from the strong caustic soda solutions.

Springing plant operation with SO_2

The operation of the springing plant itself follows closely the method of operation developed in the tar industry for the springing of carbolate liquors with flue gas.

The SO_2 gas is drawn through spray pipes set in the base of drum absorbers, the latter being built in pairs to ensure

continuous operation. The general flow diagram for this portion of the plant is set down in Fig. 1.

The sodium phenate for springing is received as a 30% solution in water containing up to 0.5% sodium sulphite in solution; care is taken, however, to exclude as far as possible any solid sodium sulphite in suspension.

It is advisable to control the springing plant operation to produce (1) the highest possible concentration of sodium sulphite solution which it is possible to process in unlagged vessels and lines and (2) a solution of sodium sulphite in which the solubility of phenol is a minimum.

It is found that these two divergent aims can best be met with a sodium sulphite solution leaving the plant at a concentration of 18% W/W, and a wet phenol concentration of 70% W/W, and to do this it is necessary to first dilute the 30% V/V sodium phenate solution from the fusion plant to 20% V/V sodium phenate solution. This dilution is performed with 10% V/V sodium phenate solution returned from the extraction plant. Any additional dilution is made with phenolic water from the distillation plant. The resulting 20% phenate solution is continually pumped to the top of a packed tower, 4 ft. diam. \times 30 ft. high, the packing consisting of $2 \times 2 \times \frac{3}{8}$ in. Raschig stoneware rings.

The phenate solution flows down

the tower counter-current to the exit SO_2 gas from the springing boilers. The gas is drawn through the tower (by a turbo blower) to remove any traces of SO_2 in the exit gas before discharging to atmosphere.

The phenate solution partially sprung in the towers then passes to the springing boiler, where the action is completed.

To reduce solubility of phenol in sulphite before separation, the reaction heat is removed from the mixture by passing it through a stirred reaction cooler. This cooler operates under pH control between the limits pH 7 to 8. If the pH value reveals that complete splitting to phenol and sulphite has not taken place, part of the solution is recirculated.

The cooled mixture of crude phenol and sodium sulphite solution is then fed to a continuous gravity settler separator.

The sodium sulphite stream from the separator is continuously decanted into a storage boiler and produces the main feed for the solvent extraction plant.

Extraction plant

In general, four methods are available for the removal of phenol from the sulphite liquors before their causticisation. They are as follows:

- (1) Steam stripping of sulphite and condensation of phenol as phenolic water.

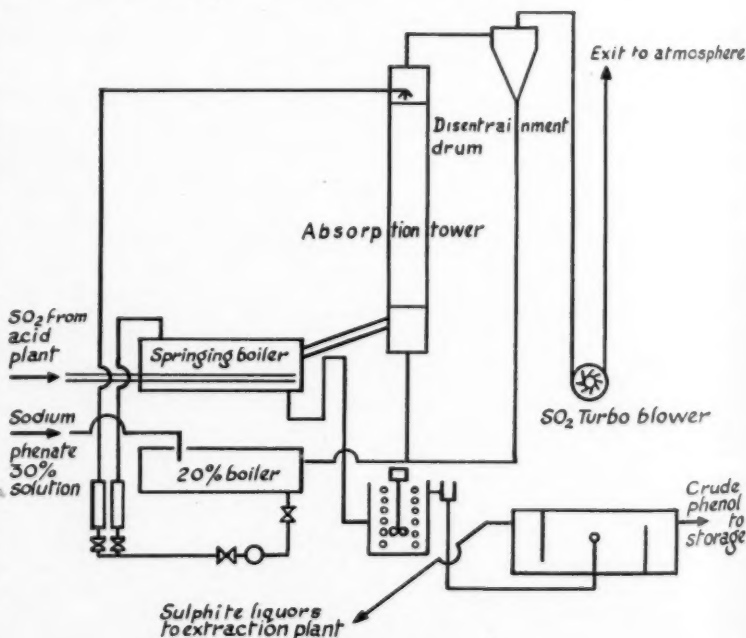


Fig. 1. Springing plant flow diagram.

larger ones, there is a critical size, and the packings for the present column must be greater than $\frac{1}{4}$ -in. Raschig rings for mass transfer to take place.

(3) The dispersed phase should be chosen so as not to wet the packing. Consequently, both for the extraction and the dephenolation tower, the benzene was chosen as the disperse phase. If the disperse phase wets the packing the liquid flows along the packing in streams rather than droplets and affects the interfacial area of contact of the phases.

(4) Action must be taken of the chemical action taking place in the dephenolation tower. This interposes a third resistance to the two-film resistances previously mentioned.

(5) Columns require introduction of perforated plates at intervals of 3 to 7 ft. for purposes of remixing phases and reducing longitudinal circulation. These plates should be designed with a few comparatively large holes so that the solvent phase which coalesces beneath the plates is effectively re-dispersed and broken down into small droplets when it re-enters the packing.

Process design of plant

The benzene and caustic extraction columns are cylindrical carbon steel vessels with a uniform diameter of 30 in. The benzene extract column is 40 ft. high and the caustic column 66 ft. Both columns are packed with $\frac{3}{4} \times \frac{3}{4} \times \frac{3}{4}$ in. random-packed stone-ware Raschig rings leaving clearance sections of 3 ft. at the top of the column and 6 ft. at the bottom with open sections for distributors. There is no enlargement of the diameter at the top or bottom of the towers since Pratt² has proved experimentally that this is unnecessary.

The solvent and feed solution must enter well in the packing and these entries are made through a hub from which perforated fingers radiate.

Cast-iron plates with longitudinal slots tapered in vertical section are fitted as distributors at intervals of approximately 7 ft. up the column. These act to inhibit longitudinal mixing and also tend to prevent interfacial crude formation. Interface control is via the usual vented syphon pipes.

Column diameter

Flooding point data may be correlated by the following expression

$$\left[1 + 0.835 \left(\frac{\rho_d}{\rho_c} \right)^{\frac{1}{2}} \left(\frac{V_d}{V_c} \right)^{\frac{1}{2}} \right] = C_2 \left[\left(\frac{V_c^2 a}{g \epsilon^3} \right) \left(\frac{\rho_c}{\rho_d} \right) \times \gamma^{\frac{1}{2}} \right]^{-1}$$

C_2 has the following values:

For Raschig rings	.. 0.68
For Lessing rings	.. 0.80
For Berl saddles	.. 0.88

It is assumed that the input is 20,000 gal./day of solution containing 18% Na_2SO_4 with 1% $\text{C}_6\text{H}_5\text{OH}$ in solution and 6% $\text{C}_6\text{H}_5\text{OH}$ in suspension. The phenol is to be reduced to a concentration of 0.05%.

Na_2SO_4 continuous-phase benzene flow rate not exceeding 70% mixed flow.

Benzene flow rate = 47,000 gal./day (Ostwald law of inversion).

Using Pratt data and a column packed with Raschig rings $\frac{3}{4} \times \frac{3}{4} \times \frac{3}{4}$ in.

a, a_v Superficial and effective superficial area of packing = 56, $56 \times \frac{3}{4}$ sq.ft./cu.ft.

g Acceleration gravity, ft./hr.²

v Superficial velocity based on empty column

ϵ Fractional voidage of packing = 0.74

γ Interfacial surface tension, = 30 dynes/cm.

ρ Density, lb./cu.ft.

$d\rho$ Density difference, lb./cu.ft.

c, d Continuous phase, dispersed phase

ρ_c = $1.175 \times 62.3 = 73$ lb./cu.ft.

ρ_d = $0.879 \times 62.3 = 55$ lb./cu.ft.

$d\rho$ = 18 lb./cu.ft.

$\frac{V_d}{V_c} = 2.35$

$$1 + 0.835 \left(\frac{55}{73} \right)^{\frac{1}{2}} (2.35)^{\frac{1}{2}} =$$

$$0.68 \left[\frac{(30)^{\frac{1}{2}} V_c^2 \times 56 \times 73}{4.18 \times 10^8 (0.74)^3 18} \right]^{-1}$$

$$\frac{4.18 \times 10^8 \times (0.74)^3 \times 18 \times (0.68)^4}{56 \times 73 \times (2.19)^4 \times (30)^{\frac{1}{2}}} = 3,760$$

$V_c = 61.5$ cu.ft./sq.ft.hr.

Operation not to exceed 50% flooding

$$\text{Tower diam. } d = \frac{2 \times 20,000 \times 4}{6.24 \times 24 \times 61.5} = 2 \text{ ft. 6 in. diam.}$$

Column height

(1) Characteristic velocity, V_o , or the mean droplet velocity relative to the packing at zero flow rates ft./hr. is determined:

(a) By initially determining a value for V_t the mean actual terminal velocity of freely rising droplets ft./hr. and from this determining the value V_o from the relation-

$$Y = 1 - \frac{Y}{Z} (1 - e^{-Z/Y})$$

$$\text{where } Y = \frac{V_o}{V_c} \text{ and } Z = \frac{d\rho g}{\rho_d V_t^2}$$

(b) By using the Pratt monograph, Fig. 2,³ and using the previously determined value of V_d and tower diameter with the recommended fractional holdup of 15 to 20%.

Characteristic droplet diameter

(2) Mean column to surface diameter of droplets at zero flow rates, ft. = d^o_{vs} .

$$\text{This is approximately } 0.92 \left(\frac{\gamma}{d\rho g} \right)$$

(3) Superficial area of contact of phases = a sq.ft./cu.ft.

$$a = \frac{6 V_d}{d^o_{vs} V_o}$$

Value of $V_o = 351$ ft./hr.; $d^o_{vs} = 2.90$ mm., where

g acceleration due to gravity, ft./hr.²

ϵ fractional voidage of packing

γ interfacial surface tension, lb./hr.²

μ viscosity, lb./hr.ft.

ρ density, lb./cu.ft.

$d\rho$ density difference of phases = $(\rho_c - \rho_d)$ lb./cu.ft.

c, d being subscripts for continuous and dispersed phases respectively.

Determination of column height

Pratt gives the following empirical relationship for k_c and k_d :

$$k_c = 0.6 V_d^{-1} d^o_{vs} V_o$$

$$k_d = 1.15 V_d^{-1} d^o_{vs} V_o$$

Data

$$\text{Dispersed phase benzene } V_d = \frac{47,000 \times 8.79}{24 \times 11 \times 62.5} \times 4 \times \frac{3}{2} = 150 \text{ cu.ft./sq.ft.hr.}$$

$$k_c = 0.6 \times (150)^{-1} \frac{2.9}{25.4 \times 12} \times 351$$

$$k_c = \frac{0.6 \times 1}{38.8} \times \frac{2.9}{25.4 \times 12} \times 351$$

$$k_c = 0.0518 \text{ lb.mols./hr.sq.ft.}$$

Interfacial area of contact = a

$$a = \frac{6 V_d}{d^o_{vs} V_o} = \frac{6 \times 150}{2.9} \times$$

$$\frac{25.4 \times 12}{351} = 269 \text{ sq.ft./cu.ft.}$$

$$k_d = \frac{1.15 \times 1}{38.8} \times \frac{2.9}{25.4 \times 12} \times 351$$

$$k_d = 0.0988 \text{ lb.mols./hr.sq.ft.}$$

$$\frac{I}{K_c} = \frac{m}{k_c} + \frac{I}{k_d} = \frac{0.25}{0.052} + \frac{I}{0.0966} = 4.53 + 10$$

$$K_c = 0.0699 \text{ lb.mols./hr.sq.ft.} = \text{overall mass transfer coefficient}$$

$$K_{ca} = 269 \times 0.0699 = 18.7 \text{ lb.mols./hr.}$$

$$m = \text{partition coefficient}$$

$$\text{concentration phenol in sulphite solution}$$

$$= \frac{\text{concentration phenol in benzene}}{0.25}$$

$$\text{Packed height} = H$$

$$W = \text{lb.mols. phenol extracted}$$

$$S = \text{cross-sectional area tower}$$

$$H = \frac{W}{S a_v K_c (\Delta c)_m}$$

$$(\Delta c)_m = \frac{0.01142}{2.3 \times 2.1461}$$

$$= \text{log mean driving force, lb.mol. units}$$

$$H = \frac{200,000 \times 6.95 \times 4 \times 2.3 \times 2.1461}{100 \times 24 \times 98 \times \pi \times 6.5 \times 18.7 \times 0.01142} = 27 \text{ ft. height of packing}$$

Caustic dephenolating tower

Chemical reaction $\text{C}_6\text{H}_5\text{OH} + \text{NaOH} = \text{C}_6\text{H}_5\text{ONa} + \text{H}_2\text{O} + 2,660 \text{ cal./mol.}$

Reaction constant $0.0014 \text{ mol./l./min.} = K$.

The effect of the additional solvent resistance is to increase the partition coefficient m .

This is increased from m to $m + 2Km^2X$, where X = concentration at the interface; then $2Km^2X = 2 \times 0.0014 \times 60 \times 0.25 \times 0.25 \times 0.1142 \times 119 = 0.143$.

$$\frac{I}{K_c} = \frac{0.393}{0.052} + \frac{I}{0.0988} = \frac{I}{17.6}$$

$$K_{ca} = 269 \times 0.057 = 15.3$$

$$(\Delta c)_m = \frac{0.0062}{2.3 \log 76}$$

$$\text{Log mean driving force} = \frac{0.0062}{2.3 \times 1.8808}$$

$$H = \frac{200,000 \times 6.95}{100 \times 24 \times 98} \times \frac{4}{\pi \times 7.5} \times \frac{1}{15.3} \times \frac{2.3 \times 1.8808}{0.0062}$$

$$= 53 \text{ ft. height of packing.}$$

$$\text{Heat of reaction} = 2,660 \text{ cal./mol.}$$

$$= \frac{200,000}{100} \times \frac{6.95}{24 \times 98} \times 1.8 \times 2,660 = 28,400 \text{ B.Th.U./hr.}$$

This amount of reaction heat will have negligible effect on the combined flow rate of continuous and dispersed phases and the corresponding rise in exit temperature can therefore be safely ignored.

Summary of design

Solvent extraction tower

18% sodium sulphite continuous phase solute phenol.

Flow rate, 20,000 gal./24 hr.

Benzene dispersed phase, 47,000 gal./24 hr.

Column diameter at 50% flooding = 30 in.

Packed height = 27 ft. of $\frac{3}{4} \times \frac{3}{4} \times \frac{3}{8}$ in. stoneware Raschig rings.

Allowing for end effect 40-ft.-tall tower erected.

Caustic dephenolation tower

10% caustic soda continuous phase flow rate, 20,000 gal./24 hr.

Benzene extract dispersed phase, 47,000 gal./24 hr.

Column diameter at 50% flooding = 30 in.

Packed height for dephenolation = 53 ft. of $\frac{3}{4} \times \frac{3}{4} \times \frac{3}{8}$ in. stoneware Raschig rings.

Allowing for end effects two 30-ft.-tall towers erected, operating in series.

Actually one 66-ft.-tall tower \times 2 ft. 6 in. diam. finally agreed.

Comments on design

The effects of the additional resistance due to the chemical reaction is to considerably increase the height of the dephenolation tower to produce the required extraction.

The height diameter ratio becomes

somewhat excessive for a single tower and led to the consideration of two towers in series.

Several reports are current on the low efficiencies generally experienced with a tower process for dephenolation with caustic soda.

With a slow limiting chemical reaction of this type the most suitable equipment has been found to be of the settler mixer type as, for example, the Holley-Mott.

It is possible that for this process the economic advantage lies with a vertical settler mixer on the lines of the Schiebel tower extractor. The indications are that such a tower would give the same number of transfer units in a height only 25% of the designed figure for the gravity flow ring-packed tower.

Due to the inflammability of benzene and its strong solvent action for hydrocarbon greases and oils, some difficulty would be experienced with the agitator shaft seal. The proposed drive would necessarily be completely flameproof, vertically mounted and driving through a water seal.

To obviate lubricating problems, all shaft bearings, including the footstep bearing, would be in lignum vitae or phenol formaldehyde plastic.

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Municipal water plants remove radioisotopes

Municipal water plants, though not designed for the purpose, can remove a substantial fraction of radioisotopes from water they process. This is indicated by studies at the Hanford atomic laboratories, Richland, U.S.A.

Mr. R. L. Junkins, a senior General Electric engineer at Hanford, reported to the nuclear congress on a year's sampling of the Pasco municipal water system filter plant 39 miles below Hanford nuclear reactors. He explained that his company, which operates Hanford for the Atomic Energy Commission, conducts this sampling as part of an extensive environmental monitoring programme in Hanford's vicinity to provide guidance in the control of plant

operation practices and to assure that the release of atomic wastes in the area is well within recognised limits.

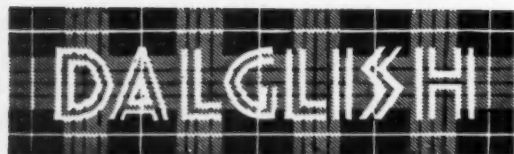
Hanford's reactors use some Columbia river-water for cooling purposes. Effluent from the reactors when discharged into the river contains several radioisotopes generated during its quick pass through the reactors. Columbia river-water at Pasco contains about 15% of the maximum concentration of radioisotopes permitted by national radiation health authorities. After treatment in the Pasco filter plant, the 15% of the permissible concentration is reduced to about five, even though this plant was not designed and is not operated for this purpose.

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Neutron Activation Analysis and Nuclear Technology

By D. F. C. Morris,* B.Sc., M.A., D.Phil., F.R.I.C.

The development of improved analytical methods for the determination of minute quantities of impurities is of considerable consequence to atomic energy technology. Impurities which are strong absorbers of neutrons cannot be tolerated in nuclear reactor fuel elements, canning materials, moderators or coolants. One of the methods providing extreme sensitivity for the determination of many elements is neutron activation analysis, which has extensively been developed in the past two decades with the establishment of nuclear reactors.

IN neutron activation analysis the sample is subjected to irradiation with neutrons for appropriate lengths of time, and chemical elements are identified and assayed by measurements of characteristic radionuclides formed from them. In some cases the activity of a desired radionuclide may be measured directly, but frequently it must be isolated by chemical separation from a large number of other induced activities. Standardisation is provided by irradiation, with the unknown sample, of a standard sample containing a known amount of the element to be analysed.¹

Most elements when irradiated with neutrons of thermal energies undergo nuclear reactions of the (n, γ) type, giving rise to radioisotopes of the irradiated element. The activity of a given radionuclide at the end of irradiation is given by the equation

$$A = \frac{f\sigma\theta W}{M} 6.023 \times 10^{23}$$

$$\left[1 - \exp. \left(-0.693 \frac{t}{T} \right) \right] \quad \dots \dots \dots (1)$$

where

A = Activity, disintegrations/sec.

f = Neutron flux, neutrons/sq. cm./sec.

W = Weight of element (g.) in the irradiated sample.

M = Atomic weight of the element.

θ = Natural abundance of the particular isotope of the element giving rise to the active species.

σ = Activation cross-section (sq. cm.) of the isotope for the particular nuclear reaction.

t = Time of irradiation.

T = Half-life of radioactive product.

After irradiation the radionuclide decays with its characteristic half-life, and at a time τ from the end of irradiation its activity A_τ is given by the relationship

$$A_\tau = A \exp. \left(\frac{-0.693 \tau}{T} \right) \dots \dots \dots (2)$$

Equations (1) and (2) suggest that W may be obtained directly, if the values of all the other parameters are known. However, there are great difficulties in such an analysis since σ is often not known with high accuracy, f is very difficult to measure exactly, and it is not always easy to determine the absolute disintegration rate A_τ . It is for these reasons that a comparative analysis is carried out with bombardment of a standard sample and the unknown under the same conditions.

Experimental

The sensitivity of neutron activation analysis is clearly dependent on the flux of neutrons, the activation cross-section, and the half-life of the particular radionuclide. High flux and high cross-section increase the sensitivity. The half-life must be favourable for measurement, and most convenient values range from a few minutes to several days.

For trace element determinations, irradiations in a nuclear reactor are generally required, and excellent facilities are available in this country. Only in cases where short-lived activities are involved does the investigator have to be in the vicinity of the

Table 1. Neutron sources available in Great Britain and usable thermal neutron fluxes (neutrons/sq.cm./sec.)

	Usable thermal neutron fluxes (neutrons/sq.cm./sec.)
Reactors:	
BEPO, Harwell ..	2×10^{13}
DIDO, Harwell ..	1×10^{14}
MERLIN, Aldermaston ..	5×10^{13}
JASON, Langley ..	1.5×10^{11}
Cyclotrons ..	10^8 to 10^{11}
Van der Graaff generators ..	10^7 to 5×10^8
Cockcroft-Walton machine ..	1×10^8
Laboratory sources:	
1 curie $\text{Sb}^{124}\text{-Be}$..	10^8 to 10^4
1 g. $\text{Ra}^{226}\text{-Be}$..	10^4 to 10^5

reactor. Samples may be sent to Harwell for irradiation in the piles BEPO and DIDO, and the Atomic Energy Research Establishment provides a radioactivation analysis service.² Other nuclear reactors which may be generally used are the A.E.I. Ltd. reactor MERLIN at Aldermaston, and the Hawker Siddeley Nuclear Power Co. Ltd. reactor JASON at Langley. The latter type of pile is now for sale at about £50,000 and can be put into operation in any part of the world. In Table 1 are shown the average thermal-neutron fluxes attainable from various sources.

Neutron activation analysis can be used for solid, liquid and gaseous samples, and usually only a few milligrammes of material are sufficient for precise work. When chemical separa-

*Brunel College of Technology, London.

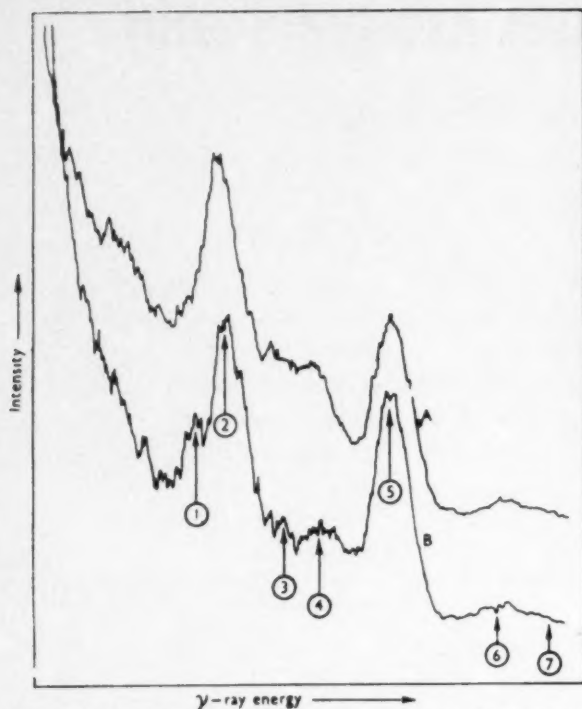


Fig. 2. Gamma-ray spectra of neutron-irradiated sodium-potassium alloy: curve A, single-crystal instrument; curve B, double-crystal instrument. (1) 0.59 MeV, caesium-134; (2) 0.65 MeV, silver-110; (3) 0.80 MeV, caesium-134; (4) 0.88 MeV, silver-110; (5) 1.09 MeV, rubidium-87; (6) 1.36 MeV, silver-110; (7) 1.48 MeV, silver-110.

From the proceedings of the Congress on Modern Analytical Chemistry in Industry. St. Andrews, 1957. Published for the Society of Analytical Chemistry by Heyfer and Sons Ltd.

tions are required to isolate the particular radioactive element, carrier chemistry is generally employed. If only micro quantities of an element in a sample are to be determined, as is usually the case, a direct quantitative separation would often be impossible. The problem is overcome by deliberately adding some inactive material isotopic with the desired radioactive product to act as a carrier in all subsequent chemical reactions. In this manner the chemical separation is transformed to a macro-chemical problem, and it is not necessary for it to be

quantitative since the yield can be measured. In most cases, normal β -counting equipment, such as a thin-window Geiger-Müller counter, may be used for the measurement of radioactivity from the sample and standard. The analytical procedure is illustrated schematically in Fig. 1.

As mentioned previously, it is not always necessary to separate the radionuclide chemically. Thus, if it emits γ -rays, these are of definite energies and may be differentiated and their intensity measured by means of a γ -scintillation spectrometer. In this

case, neutron activation provides a non-destructive method of analysis, involving only irradiation of the sample and standard and their subsequent γ -spectrometric assay. The spectrum for an irradiated specimen of a sodium-potassium alloy is shown in Fig. 2; there are present spectra of Cs^{134} , Ag^{110} and Rb^{87} , equivalent to 0.1 p.p.m. of caesium, 100 p.p.m. of silver and 5 p.p.m. of rubidium.³

Additional information on the use of γ -spectrometry in analysis can be found in reports by Morrison and Cosgrove⁴ and Connally and Leboeuf.⁵

Advantages of the method

Advantages of neutron activation analysis compared with chemical methods for determining trace elements may be summarised as follows:

(a) *Sensitivity.* With the advent of high-flux atomic piles neutron activation analysis represents for many

Table 3. Neutron activation analysis of high-purity grades of aluminium. Impurity concentrations in p.p.m.

Impurity	1S grade	Super-purity grade	Zone-refined
Manganese	190.0	1.5	0.33
Copper	53.0	18.0	0.15
Gallium	150	0.3	0.22
Scandium	0.55	0.45	0.05
Cobalt	15	8.0	31
Zinc	4.0	0.6	*
Iron	2,540	174	190
Chromium	20	—	—
Dysprosium	0.5	0.11	—
Samarium	0.4	0.09	—
Lanthanum	4.8	1.8	—
Hafnium	1.1	<0.1	—
Tungsten	<0.1	—	1.0

* Not detd.

elements the ultimate in practical sensitivity. The method has an inherent advantage for the analysis of trace impurities in reactor materials since it depends for its sensitivity on high neutron absorption by elements and such elements represent objectionable impurities in reactor materials.

An indication of sensitivities attainable under ideal conditions using the Harwell pile BEPO³ is given in Table 2.

(b) *Specificity.* The identity of the radionuclide used for the determination can be confirmed by measurements of the decay and energy of its radioactivity, in addition to radiochemical separation.

(c) *Contamination.* A feature of the neutron activation method is that samples generally require little pre-treatment prior to irradiation. Since carriers are usually employed in radio-

Table 2. Sensitivities attainable by neutron-activation analysis using the Harwell pile BEPO

Elements	Graininess
Dysprosium, europium	10^{-12}
Holmium, indium, iridium, lutecium, manganese, rhenium, samarium	10^{-11} to 10^{-10}
Antimony, arsenic, bromine, copper, gallium, gold, iodine, lanthanum, palladium, scandium, sodium, praseodymium, tantalum, terbium, thulium, tungsten, ytterbium	10^{-10} to 10^{-9}
Barium, cadmium, caesium, chlorine, cobalt, erbium, gadolinium, germanium, hafnium, nickel, osmium, phosphorus, potassium, rubidium, selenium, yttrium, zinc	10^{-9} to 10^{-8}
Cerium, chromium, mercury, molybdenum, neodymium, platinum, ruthenium, silver, strontium, tellurium, thallium, tin, zirconium	10^{-8} to 10^{-7}
Bismuth, calcium, iron, sulphur, silicon	10^{-7} to 10^{-6}

Analysis sample

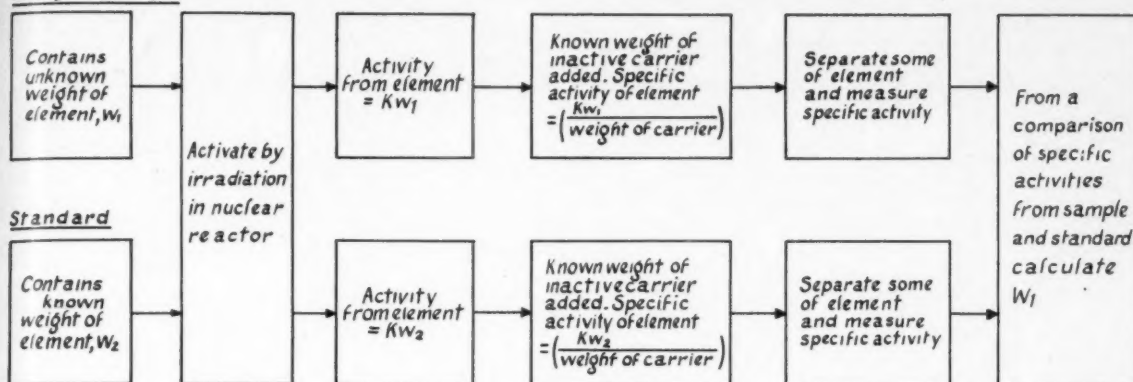


Fig. 1.

chemical separations, the method is free from difficulties caused by reagent blanks or by contamination from traces of inactive material during the chemical operations after irradiation.

(d) *Separation.* If carrier chemistry is used there is no need for radiochemical separation steps to be quantitative, as a correction is made for losses by measurement of chemical yield.

(e) *Scale of operations.* The use of carriers avoids the difficulties of chemical operations with ultramicro quantities, such as problems of co-precipitation and adsorption.

Applications

It is a requirement of all nuclear reactors that at least one of the neutrons produced in a fission process must be available for a further fission. Consequently a reactor must be as free as possible of materials which absorb neutrons in processes other than fission. This imposes severe restrictions on the nature of structural materials, moderators and coolants.

In most reactors the fuel elements are enclosed in a canning material, such as aluminium, magnesium, zirconium or niobium, which must be of high purity. Commercial grades of these materials are often unsatisfactory, for example zirconium usually contains traces of hafnium and niobium is often contaminated with tantalum. Such impurities, which have a strong tendency to capture neutrons, can only be tolerated in extremely small amounts in reactor materials, and there are difficulties attending their estimation by chemical means. Neutron activation has provided an excellent solution to the analytical problem. Some analyses of high-purity grades of aluminium⁶ are shown in Table 3.

In most reactors operating at the

present time neutrons of thermal energies are used for the propagation of the chain reaction. Such reactors must contain moderators to slow down the neutrons produced in fission. Moderators, usually graphite, heavy water or ordinary water, must be of high purity and neutron activation analysis is important in the evaluation of such materials. For example, at Harwell the method has been applied to the determination of individual rare earths and vanadium in graphite.⁷

Application of neutron activation analysis to a possible reactor coolant has been described by Smales.⁸ He has given details of the determination of mercury, chromium, rubidium, caesium, silver, antimony, strontium and cobalt in a sodium-potassium alloy.

Another application of neutron activation analysis has been to the determination of isotopic ratios in uranium samples. Seyfang and Smales⁹ determined the U²³⁵ content of depleted and enriched uranium by irradiating samples with neutrons and separating and assaying the radioactive barium produced by fission of the U²³⁵. By paying special attention to weighing and counting procedures, Seyfang¹⁰ has developed the procedure to the stage that the coefficient of variation on a single result is as little as $\pm 0.53\%$.

Neutron activation analysis has been applied to various other cases where the unknown element is already radioactive. In addition to extensive use for the determination of traces of thorium¹¹⁻¹³ and uranium,^{6, 14-17} the method may be employed for the determination of plutonium-239¹⁸ rather than direct α -counting.

There is a host of further applications of neutron activation analysis of consequence to nuclear development. The method is of importance in the

investigation of corrosion, particle size distribution and effluents. Other fields in which the technique has proved of great value are geochemistry, biology and solid-state physics.

A bibliography on radioactivation analysis up to 1957 has been compiled by the Atomic Energy Research Establishment, Harwell,¹⁹ some more recent articles of interest are listed among the references given below.

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(Concluded on page 246)

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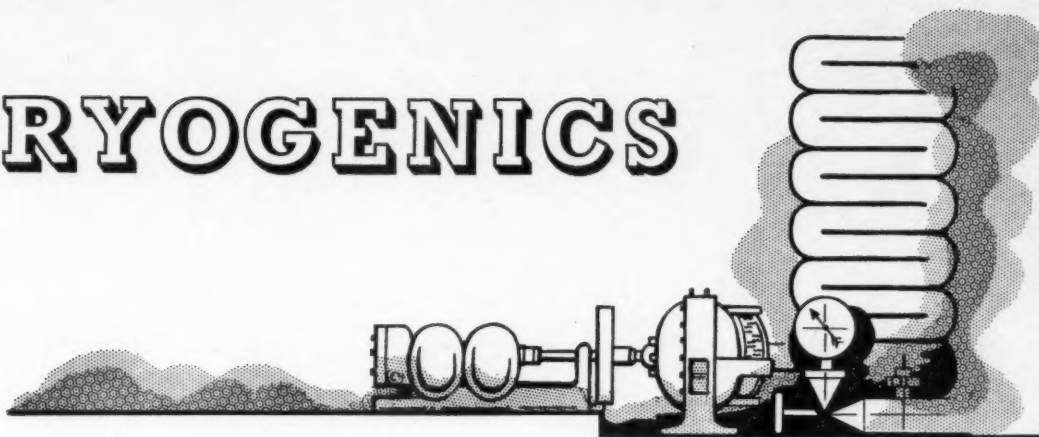
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CRYOGENICS



Low-temperature Research and Engineering

By K. Mendelssohn,* F.R.S.

Cryogenics as a major branch of applied science dates back less than 20 years. The attainment of temperatures very close to absolute zero has opened the field to interesting applications involving superconductivity. Dr. Mendelssohn, who is one of the world's leading authorities on low-temperature physics, surveys developments in cryogenic engineering.

THE history of low-temperature research begins in 1877 with the liquefaction of oxygen, the first of the 'permanent' gases to be obtained in liquid form. A few years earlier, Andrews' work on the carbon dioxide isotherms had elucidated the significance of the critical point and the subsequent development of cryogenics came as a logical sequence of these pioneer experiments. This early history, together with Nernst's enunciation of the third law of thermodynamics as the zero point of entropy at absolute zero, established cryogenics as a branch of physical chemistry. It was, therefore, in this field that the first technological applications of low temperature were to be found, and the much wider range of engineering problems to which cryogenics is directed today is a relatively recent development.

Until the late 1940's temperatures below those of liquid air were obtainable in only a few specialised laboratories and it is not generally realised that before 1930 helium gas was in very short supply and extremely ex-

pensive. However, although cryogenic engineering is a very young subject, it is growing so rapidly that a general survey becomes more difficult every year. While the account given in the present article deals with some of the more important aspects of the field, it can by no means claim to be complete.

Gas liquefaction

Liquid oxygen is produced in very large quantities but until recently the liquid phase itself was of interest only because it permits economic storage and transport of oxygen gas. The cryogenic process is, of course, necessary for the extraction from the atmospheric air. The large-scale liquefaction of oxygen for welding and steel-making is, however, a later development and it is interesting that originally there was a greater demand for nitrogen than for oxygen plants. Moreover, the first application of liquid oxygen, to be used in conjunction with carbon dust as a mining explosive, has been almost completely discontinued. It is now only being used in

certain countries where an explosive which cannot be stored or hoarded is considered to be of distinct advantage.

Since the war liquid oxygen has become important as a missile propellant and this new technology has made great demands on cryogenic engineering in the design not only of containers but, above all, of reliable handling equipment for liquefied gases. These difficulties are further increased when more exotic fuels, such as liquid fluorine, are used.

Liquid hydrogen has certainly considerable potentialities as a propellant but it is equally clear that the decrease by four-fifths of the absolute temperature from that of liquid oxygen will bring some quite new technical problems. In this region energy quantisation has become the dominant feature of the thermal properties of materials and the heat capacity as well as the latent heat of hydrogen is much smaller than that of oxygen.

Heat influx into storage tanks, service lines and handling components

*Clarendon Laboratory, Oxford.

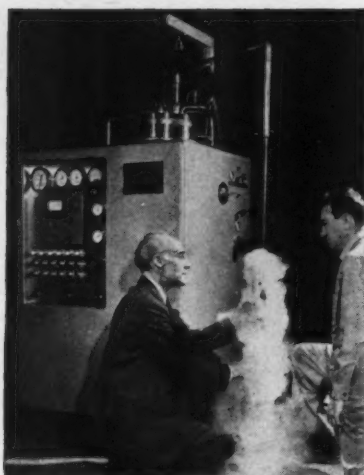


Fig. 1. A commercially produced helium liquefier designed by Prof. S. C. Collins and manufactured by A. D. Little Inc.

of a magnitude which is negligible for oxygen or fluorine can be prohibitive in the case of liquid hydrogen. In addition, the boiling point of hydrogen (20°K.) is well below the triple points of oxygen and nitrogen which means that insufficient thermal shielding will not only result in the excessive evaporation of hydrogen but also in condensation of, and possible fouling by, solid air.

Uses of liquid hydrogen

Hundreds of litres of liquid hydrogen are now being used for the investigation of nuclear reactions produced by the fast particles from large accelerating machines. Similar to the well-known cloud chamber, tracks of ionising particles can also be observed as a string of small bubbles in liquids which have become superheated by sudden expansion. Hydrogen, deuterium and helium are particularly suitable for use in these 'bubble chambers' since nuclear events in them are of special interest. Owing to the higher density and to more rapid repetition of the expansion cycle, bubble chambers are much superior to the ordinary cloud chamber. Hydrogen bubble chambers of 500 l. content have now been operated successfully, and this can be regarded as an outstanding achievement of cryogenic engineering.

Another use of liquid hydrogen is suggested by the low-temperature separation of deuterium which is required for nuclear reactions. Heavy water can be obtained by a variety of methods such as rectification of water or isotope exchange in electrolysis. Which process is the most economical

seems so far to depend much on the particular local conditions. However, separation of liquid hydrogen offers very tempting prospects in power economy, plant cost and equilibrium time, and separation on an industrial scale is being undertaken in the U.S.S.R.

At higher temperatures but on a far larger scale are the problems which arise in the shipping of liquefied natural gas. Some time ago the *Merchant Venturer*, a ship converted into a tanker for liquid methane, completed its first successful voyage. So far little technical detail concerning this important development has been made public, but it is clear that, should the transport of natural gas in liquid form turn out to be economically advantageous, a new field for cryogenic engineering will open.

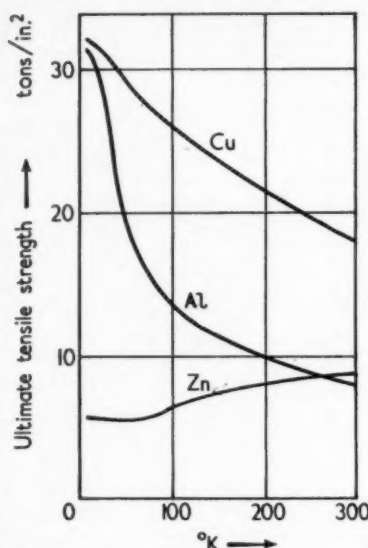


Fig. 2. Temperature dependence of ultimate tensile strength of three metals.

Insulation

As soon as the handling of liquefied gases began to exceed gallon quantities, the problem of storage had to be approached anew. Several hundred litres can, if necessary, still be stored in large vacuum flasks of special construction. Beyond this, other means of insulation have to be devised as the conventional vacuum tank becomes too cumbersome. Since the stored quantity grows with the cube and the heat influx only with the square of the linear dimensions, the percentage loss becomes less serious as the container size increases, especially if, as in the case of oxygen, the liquid phase is only required for easy transport and the time of its existence is not too long, appreciable evaporation losses can be tolerated. In these cases double-walled, lagged containers usually repay these losses by simplicity and ruggedness.

Such heat influx becomes, however, a very serious consideration when storage over longer periods is contemplated or, particularly, when the stored refrigerant is hydrogen or helium. In these cases the small heat of evaporation requires a type of insulation which is of the order of a good vacuum vessel and even these do not offer satisfactory insulation unless a radiation shield cooled with liquid air or nitrogen is interposed between the refrigerant and room temperature.

Here, technical development has been extremely successful. Insulation corresponding to a good shielded vacuum vessel has been achieved by filling the space between the room temperature shell and that holding the refrigerant with powder and evacuating it. The powder provides mechanical stability for the inner shell and, since heat transfer from grain to grain is poor, acts as an insulant.

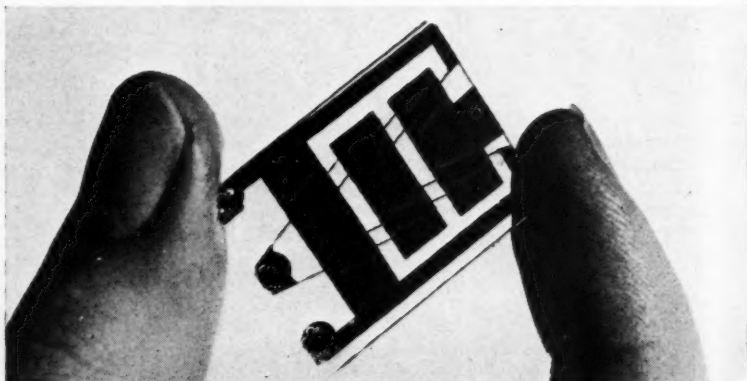


Fig. 5. A 'printed' six-cryotron circuit made by evaporation in the A. D. Little laboratories.

Moreover, since heat transfer by means of the gas residue is inhibited through the subdivision of the vacuum space by powder grains, the vacuum requirements are not nearly as rigorous as in the conventional type of Dewar vessel. The powder when cooled absorbs gas very well and thus acts as a getter. Further improvement can be obtained by imbedding in the powder a series of reflecting radiation shields. Since these are held mechanically by the powder in the same manner as the inner shell they do not present the same constructional problem as in an ordinary Dewar vessel.

Liquid hydrogen which is to be employed as coolant should, for reasons of efficiency, be turned into 100% para in the liquefier while this will be a waste in the case of liquid hydrogen fuel in a missile which is intended not to spend more than a few hours in the liquid phase. It is equally clear that there is not much point in making an attempt in this case at providing insulation which cuts down evaporation to a lower order of magnitude than that due to the ortho-para conversion.

The development of cryogenic engineering has naturally focused atten-

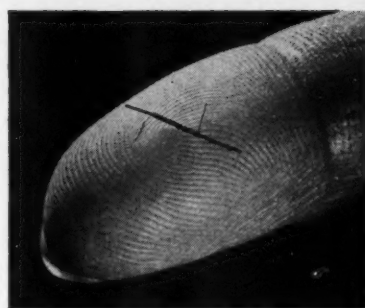


Fig. 4. Prototype cryotron.

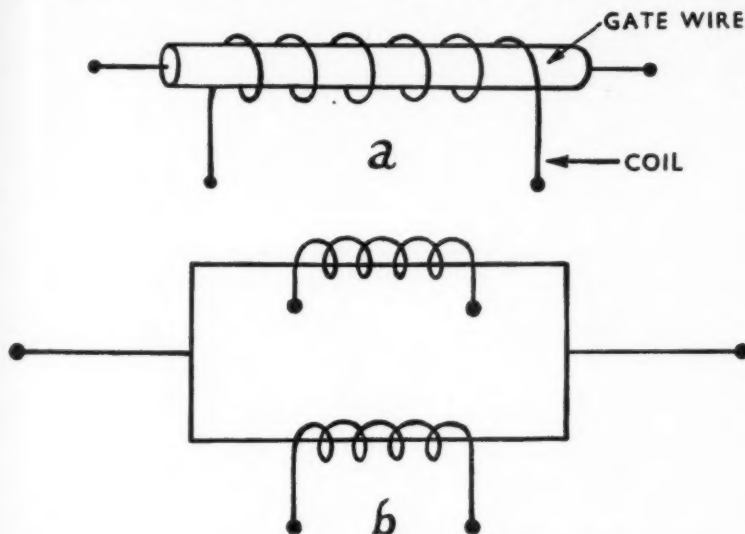


Fig. 3. The cryotron, (a) as a computer element and (b) as a memory device. The two coils serve for writing in and reading out information.

Ortho-para equilibrium

An interesting problem arises in the case of liquid hydrogen. The gas at room temperature has an equilibrium concentration of 75% of the ortho modification while at the boiling point 100% para-hydrogen is stable. The ortho-para conversion is a slow exothermic process which in freshly liquefied hydrogen of room temperature concentration leads to a noticeably increased evaporation from the storage tank. Low-temperature equilibrium can be achieved in the liquefier by providing a suitable catalyst at the cold end. On the other hand, the liquefaction efficiency is then decreased by the amount of the conversion heat. Without catalyst, equilibrium is only attained after several days and the decision whether or not to use catalysis in the liquefier must entirely depend on the time for which the hydrogen is meant to stay in the liquid phase and on its proposed use.

tion on the constructional properties of materials and particularly on metals. From the research carried out so far, a reasonably clear picture is emerging but much more work is needed before data which are reliable for engineering requirements are available. The behaviour of the stress-strain relationship appears to fall into two groups. Metals with face-centred cubic or hexagonal structure stay ductile down to the lowest temperatures with the ultimate tensile strength often showing a remarkable increase. Body-centred metals do not show this increase but tend to become brittle and fracture easily. This behaviour is reflected in the greatly increased resistance to fatigue break shown by the first group of metals when cooled to low temperatures. Less clear is the pattern presented by the results on internal friction which in some cases have revealed a large increase between liquid air and liquid hydrogen temperatures in

metal which had undergone cold work.

A somewhat surprising result was the observation of creep in cadmium single crystals at liquid helium temperatures. At normal temperature metal creep is strongly temperature dependent and this is explained by thermal activation of the movement of dislocation lines. The fact that the phenomenon has not entirely disappeared close to absolute zero suggests that this movement can also be activated by the quantum mechanical zero point energy. Another surprise was the observation of martensitic transformations in the alkali metals which are still going on at liquid helium temperatures.

Superconductivity

The observation, made almost half a century ago, that certain metals completely lose their electrical resistance at very low, but finite, temperatures, has led to a great many suggestions aimed at loss-free electrical machinery. Unfortunately, however, superconductivity is destroyed by fairly weak magnetic fields which in turn limit the available current densities to values much below those occurring in the average transformer. Thus, in spite of the fact that the most impressive and astonishing displays can be staged by means of persistent currents, they do not lend themselves to worth-while power saving.

The prospects are better in the field of instrumentation. A superconductive galvanometer has been found useful in the measurement of small thermoelectric forces and superconductive bolometers provide extremely sensitive infra-red detecting devices. However, these and similar developments are now overshadowed by the interest in superconductive computer elements. As mentioned above, superconductivity is destroyed by a magnetic field and, at one and the same temperature, this 'critical' field is different for different superconductive metals. For instance, this critical field is higher

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for niobium than for lead. Accordingly, superconductivity can be destroyed in a lead wire by a magnetic field produced by the current in a niobium wire which retains its superconductivity.

In its simplest form such a switching element will consist of a straight lead wire on to which is wound a single-layer coil of insulated niobium wire. Let us consider the existence of a steady current in the lead wire and of an increasing current in the niobium coil (Fig. 4). Then the lead will retain its superconductivity until the field produced by the niobium coil reaches the critical value for lead. When this occurs, superconductivity in the lead wire is destroyed and the resistance-free circuit of which the lead wire forms part is broken. The device thus acts similarly to an ordinary relay except that the switching does not take place between finite and infinite resistance but between zero and finite.

'Cryotron'

The great attraction of the 'cryotron', as it has been named, is the simplicity of the device and the fact that the computer network has zero resistance. In addition to providing an element which is capable of performing logic, superconductors can also be used as excellent memory stores. Storage of the information used in a computer always presents a difficult problem because usually the nature of the switching elements does not lend itself to this function. However, the phenomenon of zero resistance sees to it that, when a closed loop forms part of a circuit, a 'persistent' current will be left in the loop after the current in the network has fallen to zero. Such a closed ring will therefore carry the memory of the fact that there has been a current at a previous instance. This stored information can be picked up at a later stage by either passing another current through the circuit and monitoring its rise or by registering the memory in another network which is inductively coupled with the first one. Again, as in the case of the switching element, the device is extremely simple.

There are a great number of possible variants of both the switching and the memory element and a large volume of research is at present devoted to their investigation and perfection. By substituting for the original wire construction evaporated films, and sandwiches of such films, it may be possible to arrive at a design of printed circuits in which each print contains a large number of elements which thus can be

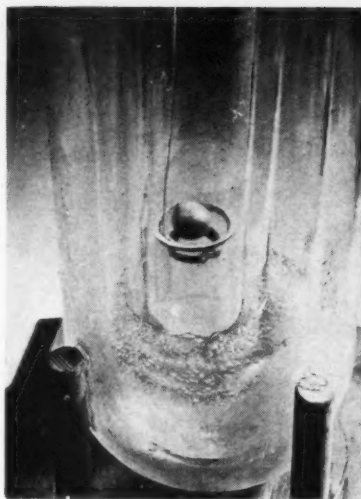


Fig. 6. A superconductive lead sphere freely suspended in a system of lead rings carrying persistent currents.

manufactured simultaneously. Here, as in most other instances of cryogenic engineering, basic and production research have to go hand in hand because, at this stage, not nearly enough is known about the fast setting up and destruction of supercurrents.

These problems involve, besides the fundamental phenomena, also the characteristic properties of the different superconductive metals in the form of thin films. Even so, the prospects for this type of computer are most encouraging. It is known that the switching speed in superconductive elements can be brought down to tens of millimicroseconds, which is quite satisfactory for a fast computer, and the type of memory elements envisaged suggest that access to the information store, too, will be fast. With these conditions fulfilled and the added advantages of compactness and negligible power consumption, the need to cool the computer to helium temperatures will not necessarily be prohibitive. The two types of work for which cryotronic computers may be particularly useful are large machines with a very extensive information store and small compact assemblies to be used in rockets whose function is fulfilled in a matter of minutes and which therefore do not require elaborate thermal insulation.

Gyroscopic devices

A still more recent suggestion for the application of superconductivity is the use in gyroscopic devices. A superconductive sphere which is dropped into a system of superconductive rings carrying persistent cur-

rents will float in space above them. As the sphere approaches the rings, their magnetic field induces a new set of currents in the surface of the sphere. The two current systems repel each other and the sphere will remain suspended above the rings at a distance at which this repulsion just counterbalances the weight of the sphere. Spinning the sphere in vacuo, we have thus a gyroscope without bearings and free from their frictional drag.

It is thought that a compass operating on this principle will permit accurate navigation in submarines for much longer times of submergence than have so far been possible and that this gyro may also become an important navigational aid for space vehicles. It has, of course, to be remembered that the superconductive sphere remains coupled to the surrounding magnetic field and that deviations from symmetry in construction will result in restoring forces exerted on the sphere from outside.

Determination of structural faults

Superconductivity is put to more indirect use in the investigation of metallic structure. At very low temperatures heat is carried in the superconductive metal almost exclusively by vibrations of the crystal lattice, the so-called phonons. These are energy quanta corresponding to a fairly large wavelength. If the same metal sample is rendered normally conducting by the application of a magnetic field larger than the critical value, the heat conduction is almost entirely by electrons. The de Broglie wave of these is much shorter than that of the phonons. Consequently, while the electrons are scattered by point imperfections in the metal, such as interstitial atoms or vacancies, the phonons are quite unaffected by them. On the other hand, large-scale imperfections, as for instance dislocations, constitute severe obstacles for phonons but electrons can pass through them without being scattered. Thus measurement of the heat conduction of a particular metal sample in the superconductive and in the normal state permits a distinction of the kind of structure faults occurring in the specimen.

For instance, it could be shown that cold work will create a very noticeable amount of large-scale damage but practically no local faults. Another important application of the method is in the assessment of damage received when the metal is subjected to radiation inside a nuclear reactor. This type of damage is as yet only imper-

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fectly investigated and its nature is not well understood so far. On the other hand, with the future development of nuclear technology, the behaviour of materials exposed to the conditions of a reactor is becoming a most important practical problem.

Other applications

The list of engineering problems involving low temperatures given above is far from complete. There are quite a number of fields in which cryogenic techniques seem to offer the only way of further progress and again others in which cryogenics opens up entirely new prospects. One of the latter is the storage and investigation of free radicals. Under terrestrial conditions these substances react so rapidly that very little was known about them. Such quantities as have been produced have had only a transient existence and this has precluded any thorough investigation. It has been found, however, that by depositing the newly created radicals on a surface cooled with liquid helium, macroscopic quantities can be collected and their physical properties are being studied. Much work has already been done on the optical and magnetic properties of the deposits and reactions in the solid phase have been investigated. Even so, the subject is clearly only in its beginnings and much further research will have to be devoted to this field until a reasonable

evaluation of practical applications for fuels or for other chemical reactions can be made.

In much the same state is the use of low temperatures for the production of high magnetic fields. Very high fields will be needed for the concentration of discharges to produce thermo-nuclear reactions. The obvious way to achieve this is to employ iron-free coils through which very large currents can be passed. A limit to these is set by the electrical resistance of the conducting metal. While, as was pointed out above, superconductors do not lend themselves to such devices because they cannot tolerate strong fields, the decrease with temperature of the normal electrical conductivity has also to be considered. The resistance of, say, pure copper is a hundred to a thousand times smaller at the temperatures of liquid hydrogen or liquid helium than its resistance at room temperature. Thus the prospects offered by coils cooled to very low temperatures are inviting, but again some major cryogenic engineering problems will have to be solved before devices of this kind can become reality.

An important aspect of electric circuits operating at low temperatures is the relative freedom from noise. This feature is not only of importance in the cryotrons but in a large number of other devices. One of them is the *Maser*, an amplifier in the microwave

region in which electrons are selectively pumped on to a higher energy level of a paramagnetic crystal. Incoming energy can subsequently be amplified by stimulating radiation from this overpopulated level. Here the use of low temperatures does not only lower the noise level but it is also necessary to achieve an appreciable inversion of level population.

Conclusion

From all this it will be clear that the use and management on an industrial scale of very low temperatures is a rapidly expanding subject which offers exciting possibilities to both physicist and engineer. The Soviet Union as well as the United States have established large government laboratories for the exploration of this new field. Moreover, a very large amount of work on these lines is also undertaken by industrial laboratories in America, many of which have their own helium liquefiers. Although this country has what is possibly the largest laboratory in the world devoted to fundamental research at low temperatures, its lead is not followed up on the industrial scale. On the contrary, Britain has not a single industrial laboratory operating a liquid helium plant, and we must face the very real danger that, as in other fields, this country provides in its universities the basic knowledge which is then turned to industrial use abroad.

International Symposium on Distillation

THE increasing importance of distillation as a unit operation in the chemical industry was stressed by Sir Alexander Fleck when he presented the opening address at the International Symposium on Distillation, which was held in Brighton from May 4 to 6.

To give some idea of the rate of growth of distillation, Sir Alexander mentioned that in 1935 at I.C.I. Ltd. the distillation load was estimated at 295,000 tons p.a. Soon after the war this had increased to 667,000 tons and by 1959 this had further increased to 2.6 million tons p.a.

The interest in this subject was demonstrated by the large number of delegates, 600, who came from every part of the world. Among those taking a leading part in the symposium were Prof. E. Kirschbaum (Germany), Dr. A. J. V. Underwood (U.K.) and Prof. W. K. Lewis (U.S.A.), who can probably be considered as the pioneers of chemical engineering. The subject matter presented ranged from theo-

retical considerations of mass and heat transfer and vapour-liquid equilibria to practical design data for commercial columns and studies of plate efficiencies.

The symposium was arranged as the 24th meeting of the European Federation of Chemical Engineering. The Association of British Chemical Manufacturers and the British Chemical Plant Manufacturers Association had set up a joint panel to examine all aspects of distillation and to indicate the lines which future research should follow. The two associations then suggested to the Institution of Chemical Engineers that the time was ripe for a symposium on the subject. This suggestion was welcomed by the Institution and a joint organising committee was set up under the chairmanship of Mr. C. S. Windebank.

From the papers offered, the organising committee, on the advice of the referees and in consultation with the chairman of the Institution's Publication Committee, Dr. R. Edgeworth

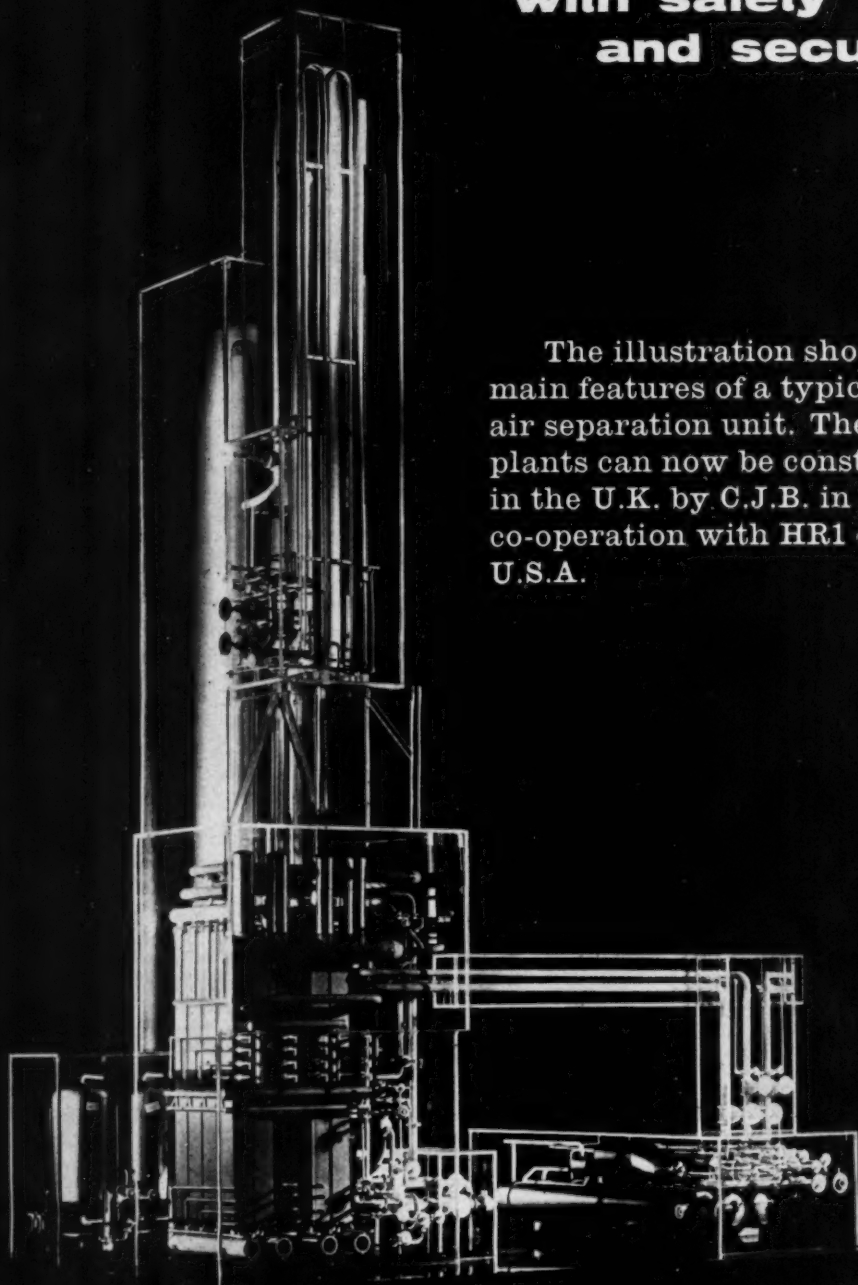
Johnstone, M.I.CHEM.E., selected 28 papers from authors in 10 countries. Pre-prints of these papers were in the hands of those registering for the symposium well in advance of the meeting. This enabled the authors to present their papers in brief and thus allow time for a comprehensive discussion.

A noticeable feature of the symposium was the simultaneous translation service into English, French and German, which was installed at great cost. This gave the meeting a truly international atmosphere, reminiscent of the United Nations. In spite of this service most German authors preferred to speak in English, whereas the Russian authors were hampered by language difficulties due to the absence of a Russian translator.

Altogether this symposium was a remarkably successful achievement and both the Institution of Chemical Engineers and all others associated with the organisation of this event are to be congratulated.

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Low-temperature Work in France

A review of academic and industrial developments in the low-temperature field in France

IT will be recalled that in 1935 Prof. Simon and Dr. Kurti, with the collaboration of French research workers, established a research group at the Centre National de la Recherche Scientifique at Bellevue which had taken measurements at temperatures lower than 1°K. This group carried out low-temperature research in various fields of physics and for several years it was the only place in France where liquid hydrogen could be obtained.

It was not until 1947 that a plant for the liquefaction of hydrogen was installed at the Grenoble laboratory, which was dependent on both the C.N.R.S. and the University. This was followed in 1952 by a helium liquefaction plant. The original aim of the centre at Grenoble was to study magnetic properties and only some time later other problems concerning solid state physics down to 10⁻²°K. were considered.

In 1956 the Commissariat à l'Energie Atomique became interested in the field of liquid helium and liquid hydrogen and established low-temperature research facilities at Saclay and, a few years later, at Grenoble.

In 1958 the C.N.R.S. decided to establish laboratories for studying the properties of liquid helium. Since then ten such laboratories have been established. Industrial laboratories, especially those attached to large electronic companies, are currently carrying out measurements down to about 1°K.

The range of the problems so tackled is obviously large and includes primarily solid-state physics. It has occurred to physicists that a convenient way of testing physical laws is to vary by a factor of 100 the temperature of the body which is being studied. Even if the quantum phenomena which appear, e.g. superfluidity, superconductivity, are of interest to only a small proportion of research workers, the consequent simplification of physical laws due to the reduction of thermal agitation is an important factor.

Application in nuclear industry

The applications of low temperatures to nuclear problems are con-

tinually increasing and only a few cases will be cited. Firstly, in connection with bubble chambers, it is interesting to note that one of 35 cm. diam. has been in use for several months at Saclay and that much larger units are now near completion. To 'feed' them more than 200 l./day of liquid hydrogen is being produced and this will soon be increased to something in the region of 1,000 l./day.

Of less quantitative importance is the work on liquid hydrogen and deuterium accelerator targets carried out at Saclay and Orsay. In the field of pile irradiation until recently, temperatures attained have not been quite as low. One such exception has been a loop of liquid hydrogen, mounted on the heavy water moderated reactor, EL3, at Saclay, for neutron moderation.

The continuing increase of openings for research has led industry and the various large research laboratories to develop new techniques. For example, over the last few months progress has been made by La Société Air Liquide in the development of new insulators in which a reduction factor of 10 in thermal conductivity has been

achieved. This has led to the conclusion that it may now be possible to store liquid hydrogen in reservoirs of several thousands of litres.

When discussing the cryogenics industry mention must be made of the place held in France by low-temperature industry. The tradition in this field was set at the beginning of the century by Georges Claude. Progress in the last few years, though it has had some bearing on the functional characteristics of plant, has mainly been directed towards the size of units constructed. Thus the *Oxylor* equipment in Lorraine has a capacity of 370 tons/day oxygen and 65 tons liquid oxygen. Other plants of similar size have been erected in France either for the production of oxygen or for the production of both oxygen and nitrogen, whilst similar plants have been constructed for installation abroad, particularly in Canada, where a total of more than 1,000 tons/day of oxygen is produced.

Another application of moderately low temperatures is for treating mixtures in coke-oven gases in order to produce ammonia. In the coal fields of Lorraine 28,000 cu.m./hr. of coke-oven gases are treated to produce 170 tons/day of ammonia. Plants of a similar capacity have been installed by French industry in Mexico, Poland, Spain and Texas.

The Commissariat à l'Energie Atomique has co-operated with industry in the construction of a heavy water plant at Toulouse. The factory at present produces 2 tons p.a. of heavy water.

Mention has not been made here of developments in plant design that units of this size entail, nor of the cryogenic applications for the purification of gases.

To conclude, attention should be drawn to the fact that this is only the initial stage in the development of low temperatures to the many problems found in the chemical and metallurgical industry. During the next few years many new applications can be expected in the field of rocket propulsion, computer memories and high-frequency oscillators.

Of interest to our readers . . .

A number of articles appearing in our associate journals this month will appeal to readers of *CHEMICAL & PROCESS ENGINEERING*.

Manufacturing Chemist—Flow Measurement, by A. Linford. *Memoirs of an Industrial Chemist*, 3: Recruiting for Research, by M. L. Burstall.

Automation Progress—Control and Handling in Modern Resistance Welding.

Dairy Engineering—Recent Developments in Aluminium Equipment, by J. C. Bailey.

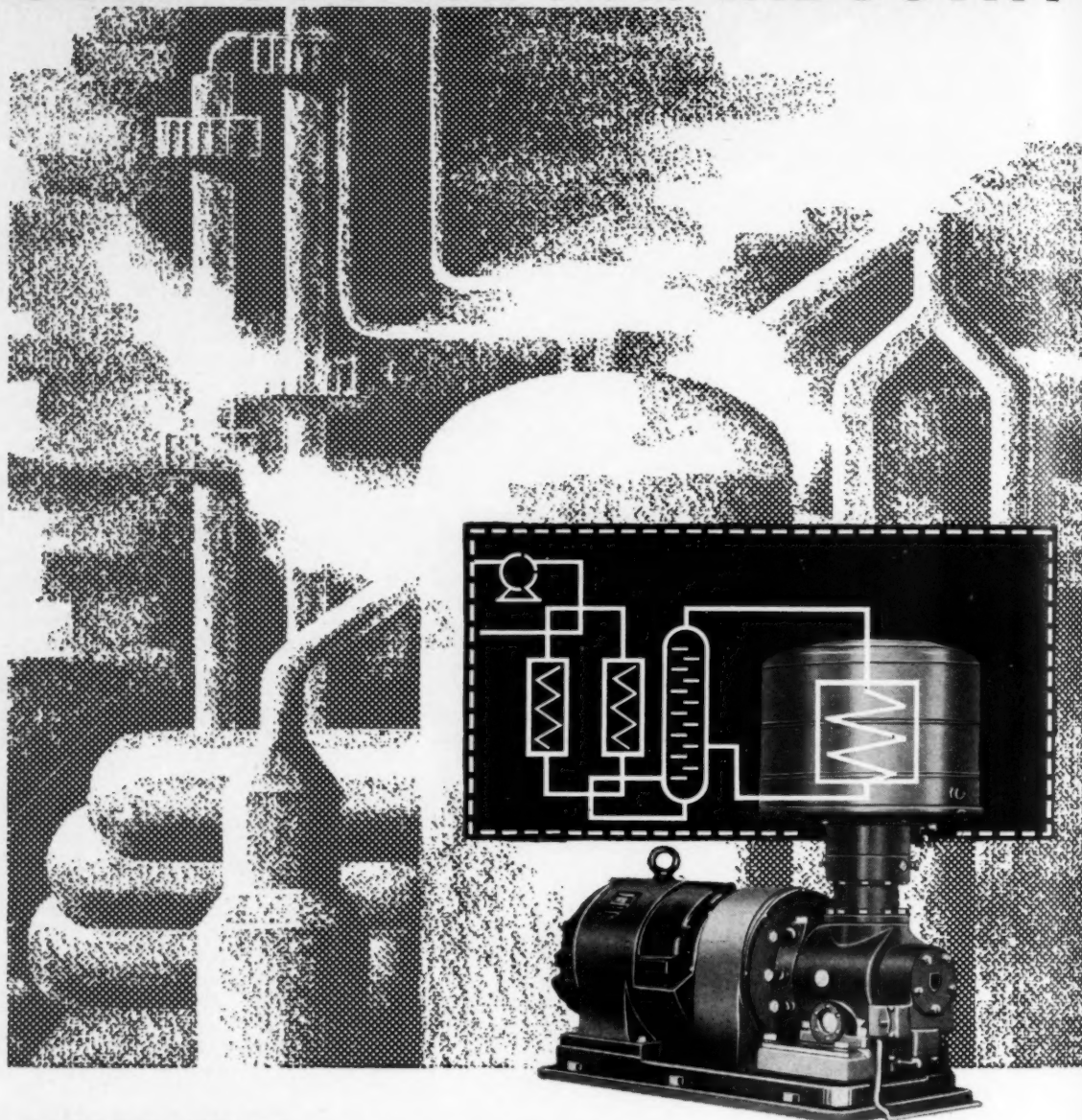
Petroleum—Natural Gasoline Recovery: Some Economic Aspects, 2.

Paint Manufacture—Paint Industries in the Outer Seven.

Food Manufacture—Special feature on Automation, review of equipment for automatic process control.

Fibres and Plastics—PVC Pastes for Coated Fabrics, by J. R. Clew.

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Tonnage Oxygen Process

By A. M. Clark,* B.A., B.Sc.

The use of large quantities of oxygen for a variety of industrial processes has markedly increased since the war. The all-important criterion of an oxygen separation plant must be its economy; this is invariably attained by careful use of heat exchange. Hydrocarbon Research Inc. has developed a highly integrated tonnage oxygen process in the U.S.A. which is fully described in this article and an estimate of its production capacity is given.

Its particular advantages are discussed.

WITHIN the last 20 years the character of many industries has been completely changed by the availability of tonnage oxygen, that is, of gaseous oxygen in massive quantities at reasonable cost. Oxygen is a fundamental raw material of most heavy chemical, gasification and metallurgical processes and when it is available only in the form of air the incubus of the accompanying nitrogen imposes severe restrictions on the efficiency, economy and technical flexibility of the basic operations in which it takes part.

The imperfections of one raw material may usually be compensated by improving the quality of others and the industries which depend on oxidation processes have been obliged in the past to rely on rich ores and high-grade fuels to offset the disadvantages arising from the relatively low oxygen content of air. Tonnage oxygen costing £4 to £6/ton or even less, according to scale and nature of demand, is today the cheapest pure industrial commodity and it has made possible the utilisation of vast resources of low-grade minerals, improved the quality of the derived products and opened up new fields of development in chemical and metallurgical technology.

Oxygen separation

The separation of oxygen from the air is accomplished by purely physical methods and the provision of oxygen at low cost depends mainly on the efficient application of thermodynamic principles. All separation processes follow a technique whereby the air is liquefied and distilled. These operations consume energy, which is introduced by compressing the air before

it enters the separation system. The more efficient the process, the less will be the energy consumed and, consequently, the lower will be the pressure to which the air must be initially compressed. Broadly speaking, therefore, an efficient air separation process must operate at a low pressure.

The energy introduced in compressing the air is expended in performing several different functions, chief among which are:

- (1) The actual separation of air into its component gases.
- (2) The achievement of the low temperatures at which the air is liquefied and fractionated.
- (3) Overcoming the resistance encountered by the air in its travel through the heat exchangers and other passages of the separation system.

The work of separation is determined by the physico-chemical characteristics of the distillation system and the energy expended in this respect is practically the same for all air separation cycles.

Maintenance of thermal equilibrium

The work expended in achieving and maintaining the necessary low operating temperature depends very much on the efficiency of the heat exchangers employed. Refrigeration at liquid air temperature requires the expenditure of considerable energy, but when the desired end-products are gaseous oxygen and nitrogen at ordinary temperatures, refrigeration becomes only a means to an end and every attempt is made, by efficient heat exchange between the in-going air and emerging gases, to retain the

'cold' which has been built up within the system. Once the system has been cooled to its low operating temperature, therefore, only a small but important amount of refrigeration must be provided to maintain its thermal equilibrium. This refrigerating effect is produced by expanding a portion of the compressed air or separation products in a suitable manner whereby the gas is made to perform external work at the expense of its contained heat.

Very large volumes of air must be processed in the production of oxygen in tonnage quantities and if refrigeration losses are to be minimised the heat exchangers necessary for the purpose must not only be highly efficient but must also afford very little resistance to the air flow if unnecessary work is to be avoided.

One practical difficulty is common to all methods of air separation. Air contains small amounts of moisture, carbon dioxide and other impurities, which must be removed before the air reaches liquefaction temperature, otherwise they would condense as solids and choke the intricate passages of the apparatus. In former practice these impurities were removed by chemical methods, which are expensive, particularly when the air is at a low pressure, and tonnage oxygen production therefore demands simpler means.

This discussion suggests that production of gaseous oxygen on a tonnage scale at low cost must be accomplished by a low-pressure process and the particular problems which have to be solved in realising this aim lie in

*Constructors John Brown Ltd., London

providing efficient means for:

- (1) Purification of the air.
- (2) Heat exchange between low-pressure gas streams.
- (3) Refrigeration at liquid air temperature.

'Linde-Frankl' process

Gaseous oxygen in tonnage quantities was first produced by the *Linde-Frankl* process about 30 years ago. This process depends on the use of regenerators, or cold accumulators, as a means of purification and heat exchange. The impurities from the air are deposited on the regenerator packing in the process of cooling by heat exchange and re-evaporated into the product streams when the flows through the regenerators are reversed. Refrigeration is effected by passing a portion of the cold compressed air through an expansion turbine whereby it performs external work. The turbine provides all the refrigeration necessary to cool the system initially and thereafter to maintain its thermal equilibrium.

More recently developed tonnage oxygen processes follow much the same general principles as the *Linde-Frankl* process, but frequently differ from it considerably in matters of engineering design. The description of the H.R.I. process which follows shows particularly how modern engineering techniques have contributed to improve the efficiency, safety and flexibility of the air separation process.

H.R.I. process

The H.R.I. process for tonnage oxygen was developed in the U.S.A. by Hydrocarbon Research Inc. and was first put into practice in two units erected in 1949 for Carthage Hydrocol at Brownsville, Texas. These units, each yielding 1,000 tons/day of oxygen, remain the largest ever built. Since then many other plants of this type, varying in size and purpose, have been built in the U.S.A. and elsewhere. Arrangements have recently been made for the complete engineering and manufacture of plants to H.R.I. designs in the U.K. and they are now available either to deliver gas 'over the fence' to steelworks and others who prefer to regard the supply of oxygen as an extraneous service, or to be incorporated as an integral part in a complete gasification or chemical process.

The process varies in detail according to the nature of the demand. Oxygen and nitrogen may be obtained at any required purity and pressure. By addition of minor attachments

argon may also be recovered when required. For some purposes it is an advantage to accumulate a store of liquid oxygen for use as a strategic reserve and by the addition of a supplementary refrigeration cycle of the necessary size any required proportion of the oxygen output may be recovered in this form. By these means the diverse requirements of the many tonnage-oxygen consuming industries may be satisfied in the most efficient manner.

Flow diagram

The simplified flow diagram (Fig. 1) illustrates a typical H.R.I. cycle. This particular cycle shows the basic design of a relatively large tonnage plant, in the range from 100 to 500 tons/day of oxygen. Modifications of this cycle are available for smaller plants and for plants that produce simultaneously with tonnage oxygen, or in any desired combination, high-purity welding-grade or medicinal oxygen, liquid oxygen, oxygen-free nitrogen, argon, krypton and xenon.

The cycle is relatively simple but highly integrated. Atmospheric air is first filtered (1) to remove dust and fog, then compressed (2) to approximately 80 p.s.i.g., cooled (3) in exchange with water, and freed of liquid water in a combination surge drum and entrainment-separator (4). The cooled compressed air is refrigerated to -278°F. in reversing exchangers (5) and (6), as it flows counter to the cold nitrogen and oxygen. All water vapour and essentially all carbon dioxide are precipitated in the reversing exchangers coincidentally with the cooling of the air.

The refrigerated air enters the base of the high-pressure column (7). There the bulk of the air is liquefied and fractionated at 68 p.s.i.g. into two approximately equal parts—an overhead liquid termed 'nitrogen reflux' and a bottom liquid termed 'rich air'. Vapour rising through the column is washed by the liquid flowing over the lower trays. This collects the last traces of carbon dioxide and any traces of acetylene.

Nitrogen, equal in volume to 20 to 25% of the in-going air, is withdrawn from the top of the column. This nitrogen fraction is then reheated to -245°F. by passing a portion of it back through the cold end of the reversing exchanger (6) and mixing this portion with the remainder (8). The reheated nitrogen is expanded to approximately 5 p.s.i.g. through a turbo-expander (9) to supply the

supplementary refrigeration requirement of the system.

Low-pressure column

Expanded nitrogen, at -315°F. , joins the nitrogen vapour stream leaving the top of the low-pressure column (10). The nitrogen reflux from the top of the high-pressure column, free of all traces of fouling compounds, is subcooled to -308°F. by exchange (11) with nitrogen product leaving the top of the low-pressure column, and then expanded through a valve (12) into the top of the low-pressure column. The rich air from the bottom of the high-pressure column is subcooled to -281°F. also by exchange (13) with the nitrogen product. It is then passed through a combination filter and absorber (14), which removes the last traces of carbon dioxide and acetylene collected from the ingoing air, and finally is expanded through a valve (15) into the low-pressure column at about the same location as the expanded air.

Between the low-pressure and high-pressure columns is the reboiler condenser (16). The rising vapours in the high-pressure column furnish heat to boil the liquid oxygen at -292°F. in the base of the low-pressure column. In furnishing this heat, the vapours are condensed at -286°F. The gaseous nitrogen product (17) leaves the top of the low-pressure column at -315°F. ; the gaseous oxygen product (18) leaves the bottom of the same column just above the liquid level.

Reversing exchanger

The oxygen product, free of carbon dioxide and water, flows at a steady rate directly from the low-pressure column through the reversing exchanger to the suction of the oxygen compressor (not shown). The nitrogen product, after exchange with the reflux streams from the high-pressure column, is further heated to -285°F. by exchange (19) with a part of the reheated air. This reduces the temperature difference between the nitrogen and the air at the cold end of the reversing exchanger to about 7°F.

The tempered nitrogen product then flows counter to the air through the reversing exchanger to the atmosphere (if it is to be wasted) or to a compressing station (if it is to be further utilised). The nitrogen product leaves the reversing exchanger at essentially atmospheric pressure and about 4°F. below the temperature of the air entering the reversing exchanger. Every 15 to 25 minutes the flow paths of nitrogen and air through the revers-

The cycle described is designed only to provide gaseous oxygen and nitrogen. When it is required to obtain a portion of the product in the liquid state a supplementary refrigeration cycle is added to the system. The working fluid for this cycle is nitrogen withdrawn from the high-pressure column and it is consequently free from all condensable impurities. After being warmed almost to ambient temperature by heat exchange the nitrogen is compressed to a much higher pressure—between 500 and 600 p.s.i.g.—in a non-lubricated compressor. It is then re-cooled by heat exchange, expanded in a high-pressure turbo-expander and returned to the high-pressure column. By this means any requisite amount of extra refrigeration can be provided without upsetting the régime of the distillation system.

The foregoing description reveals a number of features which have contributed to make the cycle safe and reliable in operation.

When the oxygen product is required at high pressure the gas is compressed in labyrinth machines which are not only safe in operation but require little maintenance.

The oxygen and, if necessary, a

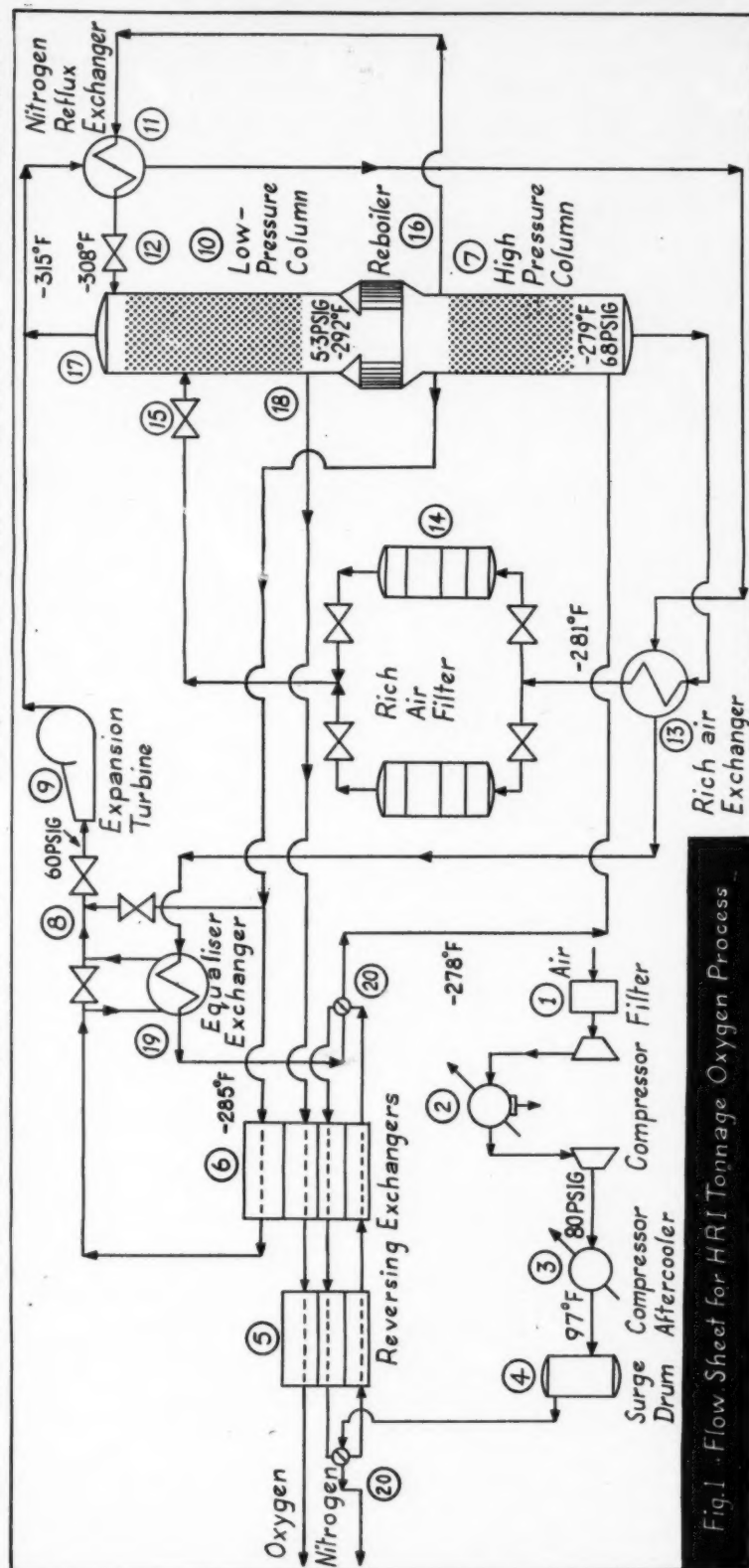


Fig.1 Flow Sheet for HRI Tonnage Oxygen Process

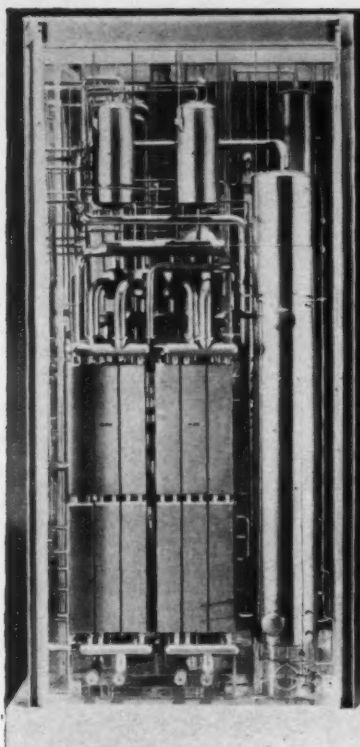


Fig. 2. Air separation unit.

portion of the nitrogen are withdrawn from the system through non-reversing passes of the exchangers, so that they take no part in picking up the deposited carbon dioxide and water vapour. These fractions may thus be obtained in a state of the highest purity.

Fig. 2 is a photograph of a model which faithfully reproduces the features of a full-scale air separation unit. Fig. 3 shows a large unit in the course of assembly. The reversing exchangers are seen in the foreground of Fig. 2 and their compact design will be noted. These exchangers are of multi-pass construction with highly extended heat-exchange surfaces. They are assembled from standard components and may be manifolded together to provide any desired combination of channels.

Materials of construction

All parts of the plant within the 'cold box' are constructed of aluminium alloy or stainless steel, metals which have excellent mechanical properties at low temperatures. All joints, except at main valves, are made by argon-arc welding so as to avoid flanges and jointing materials which might develop leaks under low-temperature stress. A method has been developed for bonding aluminium to

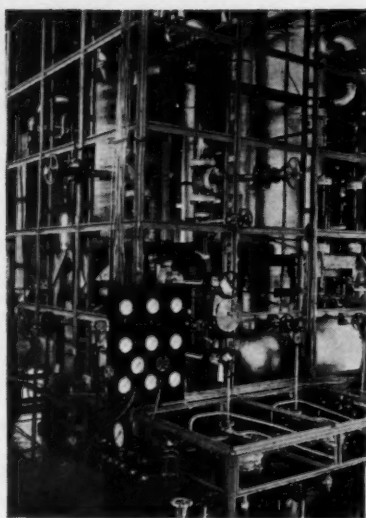


Fig. 3. Separation unit during assembly.

stainless steel when junction between these materials becomes necessary. Operating valves have spindles and packing glands extended outside the cold box. The equipment within the cold box requires practically no maintenance and the plant may be operated almost indefinitely without the necessity for de-riming.

As a result of these provisions for continuous and safe operation the need for stand-by equipment and reserve plant is practically eliminated and it becomes advantageous to install a single large unit to provide all oxygen requirements rather than to divide the load between two or more units for the sake of continuity in supply. As an example a 600-ton/day oxygen plant was installed in 1957 for the purposes of a chemical plant in Texas. Although the operation of chemical equipment having a capital value of nearly £60 million depended upon the oxygen supply only a single oxygen unit was provided and no loss of production has resulted. A number of important steelworks have also chosen to install single units to provide their highly-critical supplies of oxygen.

Neutron Activation Analysis and Nuclear Technology

(Concluded from page 231)

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- ²⁰D. H. F. Atkins and A. A. Smales, *Adv. Inorg. Chem. & Radiochem.*, 1959, 1, 315.
- ²¹G. W. Leddicotte *et al.*, 'Progr. Nuclear Energy, Ser. IX', 1959, 1, 123.
- ²²E. Merz and W. Herr, *Ibid.*, p. 137.
- ²³Y. B. Yakovlov *et al.*, *Ibid.*, p. 145.

Institute of Fuel annual luncheon

The annual luncheon of the Institute of Fuel took place at the Connaught Rooms on April 28. The Institute's Student's Medal was presented to Mr. G. E. Oatley, and the Bone-Wheeler Medal was presented to Dr. R. H. Essenhigh. The toast to the guests was proposed by the president, Dr. A. C. Monkhouse, C.B.E.

The response was made by the principal guest and speaker, Mr. Richard Wood, the Minister of Power.

Institution of Chemical Engineers—revised regulations

The regulations for admission to the classes of Graduate and Associate membership of the Institution of Chemical Engineers have recently been revised in part.

The revisions are mainly concerned with the definition of qualifications which exempt completely or in part from the Institution examination. The new basis for complete exemption from examination is the satisfactory completion of an honours degree course for a first degree in chemical engineering of a British university. The application of this principle is shown in the revisions to Appendix II of the regulations. Many who were previously required to take Part 3 (the design problem) or Part 4 (the oral examination) for promotion to Associate Membership, are now exempt from the whole examination, but will have to comply with the other requirements for such promotion.

In addition, the new edition of the regulations incorporates the revised syllabus for the engineering drawing examination, Paper D, which comes into force with the 1960 examination.

Chemical engineering scholarships

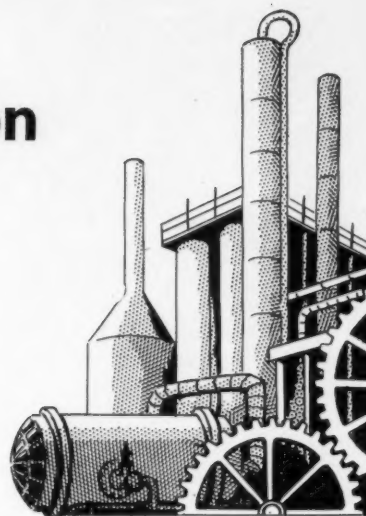
Constructors John Brown Ltd. have entered into an agreement with The Institution of Chemical Engineers for the award of two scholarships. The purpose of the scholarships is to encourage research in chemical engineering and, in particular, in chemical plant design.

The scholarships are tenable at any educational institution in the U.K. in receipt of grants from the University Grants Committee, and having a department of chemical engineering under the direction of a professor of chemical engineering.

Materials of Construction

for

Chemical Plant



LEAD

By H. C. Wesson*

M.A., B.Sc., F.R.I.C.

This is the second article in our series, 'Materials of Construction for Chemical Plant', which last month featured PVC. The purpose of this series is to discuss and evaluate the suitability of all metals and non-metals used for chemical plant construction. Throughout the ages lead was treated as a 'base' metal—whose only purpose in alchemical times was to serve as raw material for transmutation to gold. In more recent times, however, its ease of workability and chemical resistance have made it one of the most versatile metals for chemical and nuclear plant.

THERE is considerable evidence that small amounts of lead were mined in Britain in pre-Roman times, and that the inhabitants of Northern Europe were acquainted with the metal and some of its important properties, but the biggest advances in developing its applications were made by the Romans, whose extensive and elaborate plumbing systems were virtually based on lead. It could well be that their liking for the metal changed the course of history, because it has been seriously suggested that one of the reasons for Julius Caesar's invasion of these islands was to secure for the Roman Empire the lead and tin resources of Britain.

Whether or not this is true, it is certainly a fact that within a short time of their landing they were working British lead mines extensively, and many years later Hadrian built his famous wall, it is thought by some historians, not so much to keep the Scots out of England as to protect the wealthy and extensive lead mines of the Alston Moor and Newcastle districts, which the Romans were then working. Britain, as a lead-producing

country, has had its ups and downs, probably reaching maximum production in the middle of the last century, with a figure of approximately 102,000 tons p.a. of ore, which yielded about 73,000 tons of lead. Today the production of ore has declined, and probably does not exceed 2,000 tons p.a., and arises from the production from mines or quarries whose principal output is limestone, fluorspar, etc. A great deal of scrap lead is reclaimed annually—about 90,000 tons or perhaps a little more—but the rest is imported as virgin metal mainly from Australia and Canada.

Geological formation

One modern theory of the formation of metallic sulphide ore bodies is as follows:

When the earth was cooling, a hard crust formed on the outside, and as this contracted it exerted enormous pressures on the still liquid magma, compressing the centre. These pressures cracked the crust in places, and the liquid was forced up these cracks. Most metals were in the form of their sulphides, and those with high melting

points solidified at the base of the cracks, while others with lower melting points rose nearer to the surface and into the cooler regions before they, in their turn, solidified. For this reason the ore body runs more or less vertically downwards in the form of a thin sheet on edge and the metals with sulphides of low melting points, such as lead, occur near the surface. The upper portions of these have weathered with time to give carbonates, sulphates, oxy-salts, etc., but the bulk of the lead is mined today as the sulphide, galena.

When the rock containing the galena is brought to the surface, it is necessary to separate the particles of sulphide from the bulk of the material. The first step is to crush the ore to a sufficient degree of fineness for the sulphide to be physically separated from the rock in which it is embedded. The next step is to separate these two classes of particles. Modern methods depend mainly on froth flotation.

In this process the ground ore is

* Technical Manager, Lead Development Association, London.

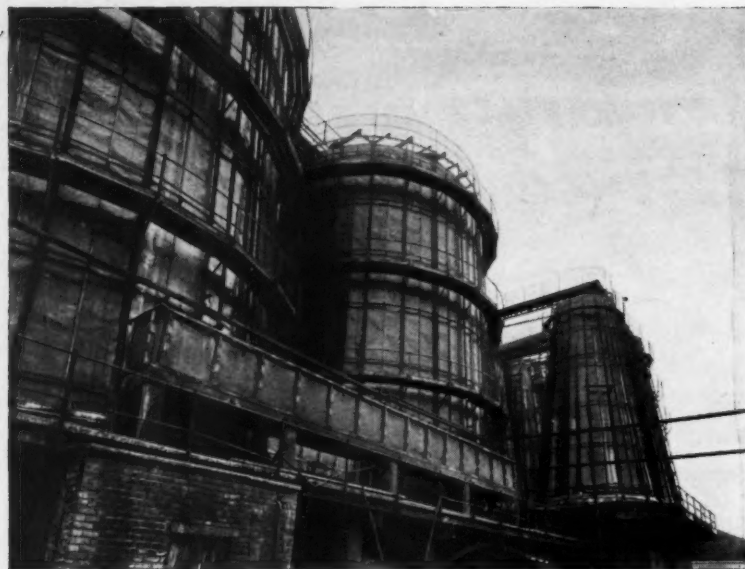


Fig. 1. Lead chambers for the production of sulphuric acid made by Mills-Packard Construction Ltd.

placed in a tank of water to which a frothing agent has been added. This may be pine oil, xanthate or some other complex thio-organic compound. The whole is agitated by stirrers and air is blown in. The air naturally rises to the top in the form of bubbles, but if the proper reagent has been used in the right concentration, the particles of sulphide ore will stick to the bubbles and be carried upwards, while the rock will remain unaffected in the bottom of the tank. The galena thus makes its appearance in a froth which is continuously skimmed off and separated by vacuum filters. By using different flotation agents, it is possible to separate one sulphide material from another, e.g. separate galena from zinc blende.

Smelting

The lead sulphide produced by this method is unsuitable for smelting in the furnace. In the first place, it contains a high percentage of sulphur, and in the second, it is too fine and would choke the air-blast. It is treated by burning to oxide on a travelling chain belt conveyor known as a *Dwight-Lloyd* machine. Suitable fluxes are mixed with the material, which is placed on a horizontal moving conveyor, to form a pad several inches thick. The sulphur dioxide produced is drawn off, cleaned and converted, usually to sulphuric acid, but sometimes to other chemicals, such as bisulphites. The product of a *Dwight-Lloyd* machine is metallic

oxide in the form of fairly coarse lumps, with a fair proportion of the smelting fluxes incorporated in this sinter. This material is then fed into the blast furnace with a small amount of coke, any further flux which may be necessary and a little scrap iron to react with any unchanged sulphide. The lead which results from this smelting operation is far from pure. It contains many other metals alloyed with it, including an appreciable quantity of silver, and sometimes a little gold. For this reason it is known as 'base bullion', or sometimes simply as 'bullion'.

Refining

There are several methods of refining, but the particular one chosen by an individual refiner depends usually on the combination of impurities in the bullion which he has to treat.

Quite a large number of metals may make their appearance as impurities in base bullion. In Group I there are copper, silver and gold; in Group II zinc and a trace of cadmium; in Group III only minute traces are found. Indium and thallium occasionally make their appearance as faint lines on a spectrograph; Group IV is represented by tin; finally, Group V by arsenic, antimony and bismuth. Of course, all these elements will not be present simultaneously in one particular sample of base bullion, and some, such as cadmium, would never be present in any appreciable quantity.

Although the refiner has several

methods available to him, all of them demand that copper shall first be removed. This is done by holding the molten lead just above its melting point, when the copper, which is insoluble at this temperature, rises to the top and is skimmed off. This is not completely efficient, and sulphur is usually added to the melt to form copper sulphide, and often a gassing agent, such as ammonium chloride, is also mixed in to cause agitation. By this means copper can be reduced to very low limits indeed. Three principal methods of refining could be referred to as fire refining, the Harris process, and electrolytic refining.

Fire refining

In the first method, when the copper has been removed, the lead is placed in a softening furnace in order to



Fig. 2. Water-cooled acid catch pot made in lead by W. G. Jenkinson Ltd.

remove arsenic, antimony and tin. These are the metals which make lead hard, and therefore the plant in which they are removed is called a 'softening unit'. The antimony, arsenic and tin are preferentially oxidised and form dross on the surface.

After softening, the bulk of the lead is next de-silvered. In order to remove the silver, advantage is taken of the fact that silver is more soluble in molten zinc than it is in molten lead, and the operation consists of washing the molten lead with molten zinc. The lead is held just above the melting point of zinc, and solid zinc is added. This melts, and is stirred into the bath of lead. When the temperature is dropped slightly the zinc begins to solidify and rises to the surface of the crust, and so can be skimmed off.

Harris process

The Harris process is used principally, though not exclusively, for refining secondary lead, because the elements which it removes, namely, antimony, arsenic, tin and zinc, are those which will be either added to lead for various uses or may contaminate the secondary lead when it is melted as scrap. In practice, caustic soda is added to a bath of molten lead when the soda melts and forms a liquid layer on the surface.

The lead is withdrawn from the bottom of the melting pot, and by means of a pump lifted and sprayed down and through the soda and into the bath once more. As the droplets of lead fall through the air, the impurities become oxidised, and these oxides are removed in the soda baths. Almost always this process is accelerated by the addition to the caustic soda layer of an oxidising agent, such as a little sodium nitrate. It is not difficult to remove any of the four elements, and a good soft lead of marketable quality can be made from industrial scrap at not too great a cost.

Electrolytic process

If lead containing both electro-negative and electro-positive impurities is electrolysed in an electrolyte containing a lead compound, pure lead only will deposit on the cathode because the electro-negative elements will go into solution and the electro-positive ones will not leave the anode as long as any lead is present. The electrolyte is lead-silico-fluoride be-

cause more common salts such as lead nitrate or lead acetate are unsuitable. Lead nitrate tends to produce lead dioxide on the anode, while lead acetate gives a very unsuitable deposit. This process was first introduced on a large scale at the beginning of this century. In more recent years the Italians have done a great deal of work in the electrolytic fields, and their preferred electrolyte is lead sulphamate in sulphamic acid.

Physical properties

Many of lead's physical properties are sufficiently well known and require only a brief mention. The metal has a density of 11.34, it is soft and malleable and can be easily worked, often by hand tools. It is resistant to a wide range of chemical reagents and welding presents no difficulty. Other properties have received less attention and may warrant a little elaboration. Ordinary lead, carrying small amounts of impurities, will recrystallise at, or a little below, room temperature. 'Type A' lead—the basis material used in chemical engineering—recrystallises at about 0°C., while material having only 1 p.p.m. impurity will recrystallise at about -50°C. There are two consequences of this which are of importance to the chemical engineer. The first is that lead will not harden by retaining cold work. On the contrary, it will remain soft and does not require any artificial annealing after it has been worked. If, for a specific application, it is necessary to employ a lead component which will harden

(e.g. in conditions of severe vibration), then a deliberate alloying addition must be made to effect this change in properties. Tellurium is the element usually chosen for this role and further reference will be made below to tellurium-lead alloys.

The other consequence of the low recrystallisation temperature involves the subject of creep. Creep may be defined as a very slow extension under stresses much lower than the ultimate tensile strength or yield stress. Most metals show this phenomenon when at temperatures above the point at which recrystallisation will take place and lead is no exception. Once the fact has been grasped that lead is in this condition at ordinary temperatures, it is easy to see that the ultimate tensile strength of the metal (about 2,300 p.s.i.) should not be the value used for design purposes.

It is considerations of creep which should determine the nature of the design and it is generally held that, at ordinary temperatures, a conservative value of 250 p.s.i. can be taken for this parameter for material which is not excessively fine-grained. The addition of tellurium has a bad effect in this connection, but fortunately a small amount of copper added to the alloy acts as a corrective and as a consequence the binary alloy tellurium-lead is not used today, being replaced instead by the ternary, tellurium-copper-lead (0.04% Te, 0.06% Cu, Balance Pb).

The comparatively high purity of 'Type A' chemical lead has obvious advantages, in that it has an excellent resistance to corrosion, but also one drawback. In common with all high-purity material it is somewhat coarse-grained, and this is a distinct disadvantage should the material be used in positions of vibration or cyclic thermal strain. The addition of a small amount of copper, about 0.07%, will produce a material with a reasonably-sized, uniform grain but with almost no sacrifice of corrosion resistance, and this alloy is extensively used in the construction of chemical plant.

B.S.334

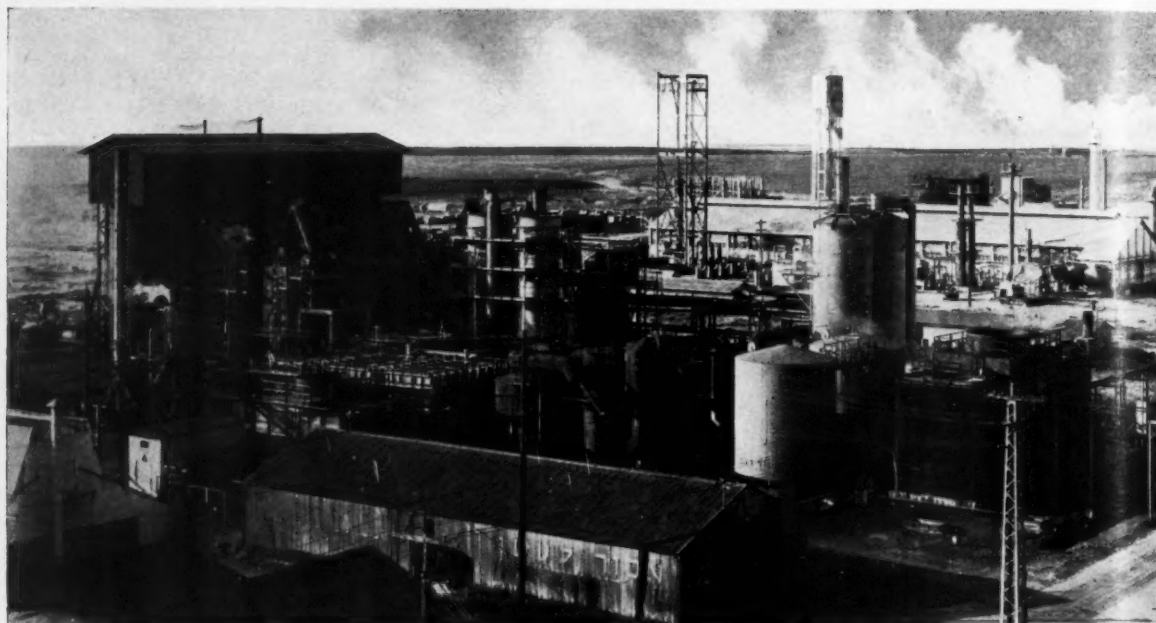
British Standard 334, 'Chemical Lead "Types A and B"', gives details of the composition and impurity limits of lead for chemical purposes, two tests and a method of analysis. Type A material is defined as containing at least 99.99% lead, while Type B is the same, but with the addition of protective elements in proportions agreed between the purchaser and manufacturer.



Fig. 3. A modern rolling mill operated by British Lead Mills Ltd.



CHEMICAL PLUMBERS



L. M. Anderson & Co. Ltd., were responsible for all lead work at this 230 ton per day Sulphuric Acid Plant in the Middle East.



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**SPECIALISTS IN
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As a corrosion-resistant material of construction in Chemical Plant, lead is still supreme. It can be economically employed with many of the chemicals used in the process industries. Examples of such chemicals are given below and a full list will gladly be supplied on request.

In addition, lead equipment can easily be repaired by cutting out damaged portions of sheets and piping and replacing with new sections, thus avoiding expensive complete replacement of equipment. Lead also has the decided advantage over other more recently developed anti-corrosion linings

in that the old lead has a recoverable value which in many cases can help considerably towards the cost of renewal.

However, the 'Burning' of lead is a skilled job and the ability to produce top-quality work only comes from long experience. This experience becomes even more valuable on site work, especially at plants overseas where facilities do not always match U.K. standards.

Our leadburners are craftsmen, experienced and versatile, and this enables us to guarantee the workmanship of any job we undertake.

Chemicals with which lead may be used

Acetone
Acetophenone
Alcohol, ethyl
Alcohol, methyl
Aluminium sulphate
Ammonia
Ammonium azide
Ammonium bifluoride
Ammonium hydroxide
Ammonium phosphate
Ammonium sulphate
Benzyl chloride
Boric acid
Cadmium sulphate
Calcium acid phosphate
Calcium bisulphite

Chlorinated polyvinyl chloride
Chromic acid
Coal tar
Copper sulphate
Dihydroxydiphenyl sulphone
Ether
Ferrous chloride
Ferrous sulphate
Hexachlorbutadiene
Hydrocyanic acid
Hydrogen chloride
Naphthalene
Nickel sulphate
Nitrocellulose
Phenol
Phosphoric acid

Phosphorus oxychloride
Pyridine
Sodium bifluoride
Sodium bisulphite
Sodium hydrosulphite
Sodium hyposulphite
Sulphur chloride
Sulphur trioxide
Sulphuric acid
Sulphurous acid
Titanium sulphate
Urea
Zinc chloride
Zinc hydrosulphite
Zinc sulphate

Alloys

Regulus metal, an alloy of lead and antimony, is the subject of B.S.335, where compositions from 6% to over 12% antimony are covered. This alloy has not the same resistance to such a wide range of reagents as chemical lead, but it is physically harder and stronger and is used in circumstances where the corrosive environment is not severe, but enhanced mechanical properties are required. Regulus metal containing more than 10% antimony is sufficiently hard to take a screw thread and can be machined and held to much closer limits than can chemical lead to B.S.334.

In order to produce a unit having the chemical resistance of lead but the mechanical properties of, say, steel or copper, chemical engineers have for many years applied lead directly to these stronger metals. They have produced thereby a material known in the trade as 'homogeneous lead',

of making homo lead have and are being devised, and it is hoped that shortly this material will be available in the U.K. in the form of sheets and pipes. It will be possible to fabricate these directly into the finished unit and all that will remain will be for the lead-burner to cover by hand the welded joints with homogeneous lead.

Corrosion resistance

In general terms, the resistance of lead depends on the fact that a surface film of corrosion product is quickly formed, and as long as this stays in close and continuous contact with the metal, no further corrosion can take place. If the environment is such as to dissolve the film, then lead is not a suitable material for the container. Similarly, if the film is not continuous but becomes cracked and is not self-sealing, an undesirable situation will result. The first condition is well known, and no chemical engineer of



Fig. 5. Remote handling of isotopes from behind a lead brick wall looking through a lead glass window at the U.K.A.E.A. Radiochemical Centre at Amersham

or more commonly, 'homo lead', which is a sheet of lead homogeneously and continuously bonded to the base metal.

Originally the steel or copper article was tinned and the metallic lead applied to this third metal layer, but it was quickly realised that this product had several shortcomings, and about 20 years ago a technique was evolved to produce homo lead with no third metal whatever. At present, homo lead is made by hand in this country and is therefore expensive, but in America this process has been mechanised, with a resultant reduction in cost. Other non-manual processes

experience would expect the lead to be satisfactory for, say, caustic soda, since the protective film of lead oxide or lead carbonate would quickly dissolve, and this action would take place progressively until the lead wall was eventually penetrated.

Attack by failure of the film's inability to remain continuous is probably more common. This is related to the softness or malleability of the metallic lead, which may allow distortion through mechanical or thermal loads. For this reason, failures in lead equipment can often be traced to physical or mechanical causes rather than to chemical corrosion, which is



Fig. 4. A large lead container for isotopes made by Associated Lead Manufacturers Ltd.

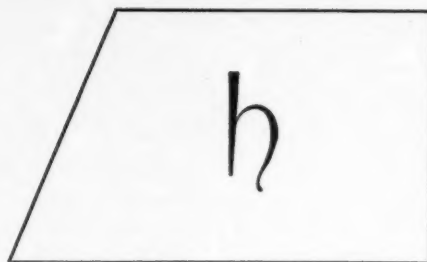
often suspected in the initial stages of the investigation. Usually attention paid to the mechanical design has been sufficient to eliminate this cause of trouble, and only in conditions of extreme severity has it been found necessary to resort to expensive homogeneous lead.

PbO₂ lining

An example may be cited which illustrates both these points. In chromium plating tanks the resistant film contains a high proportion of lead dioxide, PbO₂. This is a hard, flinty material and is formed in conditions of anodic oxidation, which can prevail intermittently on the lining of a chromium plating tank. The modern technique is deliberately to make the lining anodic for a short period when starting up a new tank, and so produce the PbO₂ film. If, however, soft lead has been used for the lining, mechanical and thermal stresses may cause it to crack and so expose the metal underneath, and unless this happens to be an anodic zone when the tank is in use, attack by chromic acid to produce lead chromate will result.

Lead chromate has not the same sealing-off properties as lead dioxide and failure of the lining may ensue. The substitution of a hard antimonial lead instead of a soft chemical lead goes a long way to eliminate this failure. The final refinement, however, is to add a minute amount of silver to the antimonial lead, because this material has been noticed to have

How much more difficult
things would be if it
were not for
LEAD



- more difficult to fabricate
- more difficult to produce economically
- more difficult to obtain immediately

In many cases lead provides the only material which
will give a completely satisfactory job



This lead filter tank is an example of the advantages of lead to the chemical engineer. It was made by J. H. Shouksmith & Sons Ltd. of York and is an example of craftsmanship in chemical plumbing. The main body of the tank is 3' 6" internal diameter and of 1' 9" maximum depth and is constructed of 20 lb. chemical lead turned over a mild steel rod at the top. The whole of the base is covered with 6 lb. chemical lead. Air is conducted through a $\frac{1}{2}$ " x 9 lb./yd. chemical lead pipe supported on rubber rings.

All the jointing of the lead is, of course, effected by leadburning.

The technical officers of the Association are always glad to give individual assistance.

LEAD DEVELOPMENT ASSOCIATION

18 ADAM STREET, LONDON, W.C.2

Telephone: WHitehall 4175

Telegrams: Leadevop, Rand, London

Table I

CORROSION RESISTANCE OF LEAD

A = Reduction in thickness less than 0.1 mm. p.a. = resistant.

B = Reduction in thickness less than 1.0 mm. p.a. = fairly resistant.

C = Reduction in thickness less than 1.0 mm. p.a. = not resistant.

	20°C.	60°C.	100°C.	140°C.		20°C.	60°C.	100°C.	140°C.
Acetaldehyde	A				Fatty acids	B ¹⁴	B ¹⁴	B ⁴	
Acetic acid	B ^{1, 2}				Fluorine	B	B		
Acetic anhydride ..	A ³	B ⁴			Formaldehyde ..	A ¹⁵	A ¹⁵		
Acetone	A				Formic acid	A ¹⁵	A ¹⁵		
Acetylene	A ⁷				Glycerine	A	A		
Aluminium chloride ..	B ³				Hydrochloric acid ..	C(B ¹⁰)			
Ammonia	A	A	A ^{6, 10}		Hydrogen bromide ..	A ^{9, 4}		B	
Ammoniumbicarbonate	A				Hydrogen cyanide ..			A ^{6, 4}	
Ammonium carbonate	A	A	A		Hydrogen fluoride ..	B ⁸			
Aqua regia	C				Naphthalene	A			
Barium carbonate ..	A				Nitric acid	B ¹⁶			
Barium sulphate ..	A				Nitrobenzene	C			
Benzene	A				Oleum	B	B	B	
Benzenesulphonic acid	B				Organic acids	C ¹⁷			
Benzoic acid	B				Oxidising gases ..	A	A	A ¹⁰	
Bromic acid	A ⁴				Petrol	A ¹⁸			
Bromine	A ^{5, 10, 11}				Phenol	B	B	B ⁸	
Calcium carbonate ..	A				Phosphoric acid ..	A	B ¹⁹	B ²⁰	
Calcium hydroxide ..	B ¹⁰				Potassium hydroxide ..	C			
Carbon dioxide	A	A	A	A	Sodium bicarbonate ..	A ³			
Carbon disulphide ..	A ⁶				Sodium bisulphate ..	A	A	A	
Carbon tetrachloride ..	A	A ¹⁰			Sodium carbonate ..	B ³	C		
Chlorine	A	A	B ¹⁰		Sodium chloride ..	B	B		
Chloroform	A	A			Sodium hydroxide ..	C			
Chlorsulphonic acid ..	B ⁶				Sulphur dioxide ..	A	A	A	A
Chromic acid	A ⁹	A ⁹	B ¹²		Sulphuric acid ..	A			
Citric acid	A	A			Sulphuric acid (fuming)	C			
Coal tar	A				Sulphurous acid ..	A			
Concrete	C ¹³				Toluene	A	A	A	
Copper sulphate ..	A ⁵	A ⁸			Town gas	A	A	A	
Cresol			B		Water (distilled) ..	A ²¹	A ²¹	A ²¹	
Ethane	A				Water (natural) ..	{ A ²² B ²³	A ²² B ²¹	A ²² B ²¹	
Ethylene	A								
Ethyl ether	A								

REFERENCES

¹Quiescent—and with air completely excluded.

²Fully resistant to cold glacial acetic acid.

³10%.

⁴With H₂SO₄.

⁵100%.

⁶Hard lead.

⁷Absence of phosgene (PH₃).

⁸60%.

⁹12%.

¹⁰Dry.

¹¹Free from acid.

¹²50%.

¹³Lead must be protected from green concrete by bitumen or asphalt.

¹⁴Absence of air.

¹⁵Resistant to 100% but not below 85%.

¹⁶60 to 80%.

¹⁷Generally not recommended. May be satisfactory if free H₂SO₄ is present.

¹⁸Sulphur must not be present.

¹⁹85%.

²⁰80%.

²¹Absence of dissolved O₂ and CO₂.

²²Hard water.

²³Very soft water.

some odd properties where lead dioxide is concerned, and will form a very strong mechanical bond between itself and this compound. In this way the protective film is held firmly by the chemical composition of the alloy, and secondly, the occurrence of cracks is kept to the minimum by using an alloy the hardness of which is nearer to the hardness of the film.

Sheet and pipe

Lead is used in many chemical industries and for many and varied pieces of plant, as shown in Table 1 and Figs. 1 and 2. Most of this equipment is fashioned from sheet and pipe, although lead castings also play a minor but important part. These components are readily available in any of the alloys used in the chemical trade, and can be supplied to almost any size.

Although the usual size of a lead sheet is 30×8 ft., sheets of up to 60×12 ft. are available, and smaller sizes are supplied on request. The thickness of sheet is defined by the weight/sq. ft. In this connection it should be remembered that 1 sq. ft. of lead, 1 in. thick, will weigh 60 lb. Thus, $\frac{1}{4}$ in. lead sheet is known as 15-lb. material, $\frac{1}{2}$ in. as 12 lb., and so on. Sheets up to 25 to 30 lb. are rolled for despatch purposes, but over 30 lb. ($\frac{1}{2}$ in.) it is delivered flat, and is known as plate. Lead sheet or plate can be made in a range of sizes from material too thin to interest a chemical engineer up to about 4 in. thick (see Fig. 3).

Lead pipe is available in almost any combination of bore and wall thickness, up to an outside diameter of about 12 in. The smaller sizes are coiled for delivery, but the heavier,

stiffer components are despatched in lengths of 14 to 16 in. and are called barrel. The conventional way of describing lead pipe is by the internal diameter in inches, followed by the weight/yd. in lb.—e.g. $1\frac{1}{2} \times 18$ lb.

Radiation shielding

Lead is extensively used for radiation shielding, principally because it is the cheapest heavy metal. There are two hazards arising in the field of nuclear energy, from gamma rays and from neutrons. Gamma rays are a form of electro-magnetic energy, very penetrating and destructive to human tissue. Neutrons, as such, are not dangerous, but when they encounter an obstacle and are stopped, they generate gamma rays, and a health hazard immediately arises.

The problem therefore resolves itself into stopping the gamma rays and stopping the neutrons, followed by absorption of the gamma rays so produced. At the energy levels now commonly encountered, say in the general neighbourhood of 1.0 MeV, the stopping power of an element depends upon the square of its atomic weight, and of those metals having high atomic weights (bismuth, lead, mercury, platinum, thallium, uranium, gold, etc.) lead is the only one whose price enables it to be used on the large scale that is required.

Radiation shields

It is comparatively easy to design lead containers for small quantities of isotopes, etc., but when considering large lead shields for reactors, two points must be remembered. If it is necessary to transmit heat through these shields, either directly from inside to the outside steel casing or indirectly from the lead shield to water-cooled copper pipes embedded in the shield, then the technique of homogeneous attachment must be used to eliminate air gaps which are such bad conductors of heat.

Secondly, if the lead units are over a certain size, then allowance must be made for the differential coefficient of thermal expansion between lead and steel. This is usually arranged by making the lower part of the lead shield to be a slightly loose fit in its steel container, so that it can expand relatively in an upward direction, and then to hang the upper part of the lead shield from the inside roof of the container, the attachment usually being made by a homogeneous bond. The two parts of the shield meet at a 'V' junction to prevent the passage of the rays.

The material of construction for chemical plant which will be discussed in next month's issue of CHEMICAL & PROCESS ENGINEERING will be

NICKEL

Fabrication of radiation shields calls for considerable skill. In common with most metals, lead shrinks when passing from the liquid to the solid state, and unless precautions are taken, a lead casting will contain a 'pipe' or shrinkage cavity.

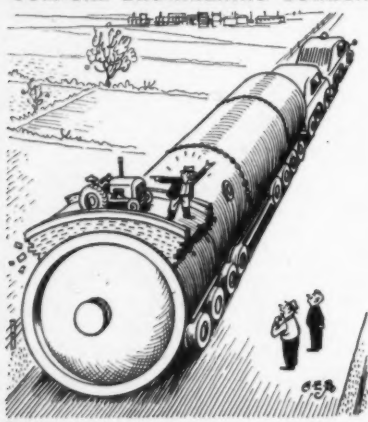
In recent times a practice has been evolved of making these thick units by cold rolling and/or multiple layers, advantage being taken of lead's extreme malleability even when cold. It is not always possible, however, for the fabricator to use that method best suited to the problem on hand, since considerations of the design do not always give him complete freedom of choice, but it is expected that as the industry grows, and each side becomes aware of the problem facing the other, these difficulties will be overcome by closer collaboration (see Figs. 4 and 5).

Economics

It has always been recognised that lead is an extremely durable material of construction and that when its useful working life is over its intrinsic value is still high, representing a substantial proportion of its original cost. Indeed, in these days of inflation the scrap value of a lead unit may be higher than the original purchase price. On account of this situation it has long been the practice of manufacturers of lead sheet and pipe to make arrangements with the owners of chemical plants whereby the cost of old lead returned is credited against new lead at a rate which is most advantageous. As a consequence, lead is one of the cheapest materials of construction on many chemical undertakings.

Chemical plant construction is now a subject of considerable complexity and it is difficult to assess accurately the value of any particular material in this field; but the consumption of lead by this industry seems to be steadily maintained year after year, and it is clear that this material is continuing to make a contribution which is as valuable today as it has been in the past.

COMICAL ENGINEERING CORNER



'I THOUGHT WE CUT IT A BIT FINE UNDER THAT BRIDGE, JOE'

Chemical Reaction Engineering

By I. L. Hepner,* Ph.D., A.C.G.I.

THE European Federation of Chemical Engineering celebrated its 25th meeting with a symposium on 'Chemical Reaction Engineering' held in Amsterdam on April 26 to 29. This subject is one of those new branches of chemical engineering which is really a hybrid between reaction kinetics and process engineering. In view of the increasing importance that chemical engineering has attained both as a science and a technology it is becoming ever-more useful to discuss new aspects of applied physical chemistry under the aegis of chemical engineering.

This was shown by the large and varied membership at the symposium, which included mathematicians, physicists, physical chemists, chemical engineers and mechanical engineers. In all, 480 delegates representing 16 countries attended. The largest delegation came, not unnaturally, from the Netherlands (192), the second largest was from Germany—both West and East—(92), the third and fourth largest delegations were Britain and France, who had 51 and 32 delegates respectively. The Soviet delegation consisted of three delegates, including G. K. Boreskov, a corresponding member of the Academy of Sciences.

Topics

The total number of papers presented was 28. The conference was divided into sessions, each one devoted to a particular topic; these included selectivity and optimisation, gas-liquid reactions, liquid-liquid reactions, fluid-solid reactions, residence time distribution and mathematical techniques and models. It was a wise decision to limit authors only to reading synopses of their papers and to leave most of the time to discussions. As a result many new points were brought to light and delegates who had been unable to write a paper themselves used the opportunity to give a résumé of work carried out on related subjects.

Prof. Kramers, in his introductory address, compared the symposium with a large reactor in which hypotheses were being converted to established theories, cherished beliefs vaporised and the general state of knowledge polymerised. It is, of course, impossible in a short report to mention all the new 'reactions' carried out at Amsterdam. Several interesting papers however will briefly be mentioned.

Micro- and macro-kinetics

L. Kuchler, in an introductory paper, stated that the sole task of reaction engineering is to obtain the best conditions under which a chemical reaction to yield a certain product can take place. Best conditions are achieved by attainment of minimal production costs for a specific rate of production. Thus the real task of chemical reaction engineering starts as soon as a process is translated from laboratory to pilot plant. Similar problems occur, of course, during improving or scaling up of existing plant, either by converting from batch to continuous processes or by installation of automatic process control. This study of macro-kinetics can be termed applied reaction engineering.

As opposed to this, pure reaction engineering deals with micro-kinetics, for instance determination of the order of reaction, and activation energy. Pure reaction engineering tries to determine the quantitative aspects of the mechanism of reaction by examination of elementary and side reactions which contribute to the main reaction. Thus studies on different levels of micro- and macro-kinetics are necessary in order to determine the conditions under which a reaction should be carried out to give a maximum yield.

Overall reaction yields

The terms 'overall' and 'instantaneous' reaction yields were discussed and defined by K. G. Denbigh. He mentioned that the reaction yield

is often less than 100% for the following reasons

- (i) Insufficient duration of reaction;
- (ii) attainment of a state of thermodynamic equilibrium;
- (iii) occurrence of wasteful side reactions.

The third point is particularly important as a cause for low yield, because the relative velocities of useful and wasteful reactions compete with one another to determine the yield. The ratio of these velocities may vary during the course of the reaction on account of the change of composition, therefore the achieved yield is most sensitive to the prevailing conditions. Denbigh described experimental methods of determining instantaneous yields, and used the results for calculating optimum reactor design both for batch and continuous stirred tank reactors.

Nitrous gas absorption

Related work concerning nitrous gas absorption was reported by H. Kramers at Delft and S. P. S. Andrew and D. Hanson at I.C.I. Billingham. Both teams investigated the reaction kinetics and mass transfer phenomena associated with absorption of nitrous gases in water. Kramers suggested that the transfer of NO_2 or N_2O_4 from an inert carrier gas to water can be explained by a diffusional transfer of the equilibrium mixture $\text{NO}_2/\text{N}_2\text{O}_4$ in the gas phase followed by preferential dissolution and diffusional transfer of N_2O_4 in the aqueous phase combined with a fairly rapid first order reaction between N_2O_4 and water. In order to obtain more accurate information on the liquid phase kinetics of the chemical absorption of N_2O_4 in water, laminar water jets of different lengths were exposed to a pure $\text{NO}_2/\text{N}_2\text{O}_4$ mixture at various pressures.

The amounts of HNO_2 and HNO_3

*Editor, CHEMICAL & PROCESS ENGINEERING

found by chemical analysis in the liquid leaving the absorption system were interpreted in terms of absorption of N_2O_4 and its subsequent reaction. It was shown that N_2O_4 is preferentially absorbed under simultaneous reaction with water according to a fairly rapid first order formula. A sketch of the absorption apparatus incorporating the jet is shown in Fig. 1. Andrew and Hanson started off from the same premises and interpreted the dynamics of the absorption of nitrous gases in water as a result of a number of separate reaction mechanisms. They pointed out particularly that the relative importance of these mechanisms is primarily dependent on the gas composition. Thus at high gas concentrations the predominant absorption mechanism is dependent on the NO/NO_2 ratio in the gas phase. When this ratio is less than 0.5 the predominant mechanism is the liquid film limited solution of NO_2 . When the ratio is bigger than 5 the predominant mechanism is the absorption of HNO_2 followed by its homogeneous liquid phase decomposition. At all intermediate concentrations, however, more than one absorption mechanism is of importance.

Kinetics of oil-sweetening

The Shell air-soluliser sweetening process was described by H. Groothuis. This process converts malodorous mercaptans present in certain gasoline fractions into less pungent disulphides by contacting gasoline in which oxygen has been dissolved, with an aqueous solution of potassium hydroxide and certain organic compounds like fatty or cresylic acids known as 'solulisers'. Fig. 2 shows the flow scheme of the essential part of the process. In the reactor the following steps must be distinguished:

1. Transfer of mercaptans and oxygen from the gasoline to the soluliser phase.
2. Reaction of mercaptans and oxygen in this phase to disulphides and water according to $4RSH + O_2 \rightarrow 2RSSR + 2H_2O$.
3. Transfer of disulphides from the soluliser phase to the gasoline.

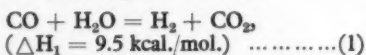
As the chemical reaction is irreversible the concentration of disulphides and the rate of their transfer to the gasoline will not influence the overall rate of conversion. Hence the process can be described by the mass-transfer equations for mercaptans and oxygen together with the material balance and equation for the chemical reaction. The heterogeneous reaction

between oxygen and mercaptans was found to be first order in the oxygen concentration and of the order of 0.25 in the mercaptans concentration. Groothuis concluded from these data that even at a stirring energy as high as 5 h.p./cu.m. the overall rate of conversion is still limited by the rate of oxygen transfer.

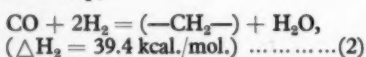
Kolbel-Engelhard synthesis

The synthesis of liquid hydrocarbons from gases has occupied German research workers for decades. The Fischer-Tropsch synthesis, although technologically interesting, was doomed to failure due to the low cost of natural petroleum. A similar type of synthesis was achieved by Kolbel and Engelhard in 1948 and H. Hammer described some of the reaction kinetics in a fixed-bed and gas-bubble reactor during such a reaction. The overall reaction can be shown as follows: $3CO + H_2O = (-CH_2-) + 2CO_2$ where $(-CH_2-)$ is one unit of a hydrocarbon molecule. The catalyst required can be selected from Group VIII of the Periodic Table.

It was found desirable to level up the intermediate stationary maxima of the partial pressure of hydrogen observed in kinetic studies in fixed-bed tests in order to load the catalyst more equally and to suppress the undesired formation of methane. This was achieved with a remarkable success in a gas-bubble column reactor with finely divided catalyst suspended in synthetic liquid hydrocarbons. The reactor is shown in Fig. 3. The gas enters the reactor at the bottom inlet and comes into contact with liquid synthetic hydrocarbons, and leaves the reactor at the top outlet. The liquid slurry serves both as carrier for suspended catalyst and heat exchanger. Kolbel and his workers found that there is a two-step reaction involved in this process. At the surface of the catalyst, adsorbed CO reacts with adsorbed H_2O as follows:



the actual synthesis takes place in the next step,



The water formed in the reaction (2) is used in reaction (1). The advantage of this continuous process over the Fischer-Tropsch process was shown by the lower H_2 partial pressure, less methane formation and a consequently better distribution spectrum.

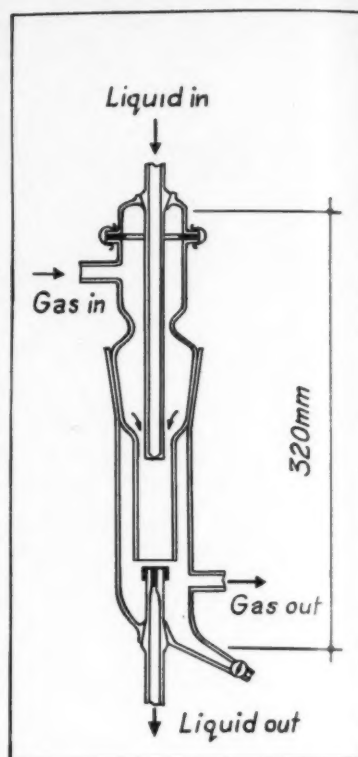


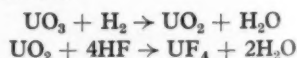
Fig. 1.

Ion-exchange plant

It is surprising that there is no theoretical basis for calculating ion-exchange plant as was reported by E. H. Becker-Boost. This deficiency applies particularly to systems using solutions of higher concentrations and ion-exchangers with a higher degree of cross linking than the usual commercial products.

Gas-solid applications in uranium processing

A. R. Cooper and J. E. Lloyd described some reaction kinetics involving uranium tetrafluoride. This, of course, is a key intermediate in the production of uranium metal and of uranium hexafluoride. The production of uranium tetrafluoride can be shown as:



The rate of these reactions has been shown to be determined by the structure of the solid surface which is modified in turn by the progress of the reactions. The thermal damage phenomena which have been observed are of greater significance in this type of heterogeneous reaction than in the more general fields of combus-

tion and catalysis. These influence the choice, design and operation of an ideal reactor system for the production of UF_4 .

Simulation techniques

In one of the best British contributions to the symposium, P. V. Youle described simulation techniques which are capable of making design-work more effective and of bridging the many gaps between scaling up of a chemical plant from laboratory to full-scale operations. He described techniques using statistics and an electronic computer to generate a comprehensive model of the full-scale plant.

This model is built up in stages, and each laboratory experiment is planned to give a simulation of the chemical reacting system. To this is added, at the semi-technical stage, a set of terms based on the relevant scale dependent

the discussion came from universities, government research laboratories and large industrial concerns. This, of course, is another proof that first-class fundamental and applied research is only carried out nowadays in such organisations.

The largest number of papers was presented by the Dutch and the Germans with the British and French some way behind.

Qualitatively speaking, the Dutch and German papers were of the first order and it was markedly noticeable that the schools at Delft, Darmstadt and Berlin are at the moment carrying out superb research work in large teams. Prof. Schoenemann, for example, has been working on kinetics at Darmstadt for close on 30 years. It is not surprising, therefore, that reaction kinetics are so well understood and applied in Germany and Holland.

The best summary of the symposium

establishment of the Common Market and consequent economic unification of six European countries, Germany has assumed a dominant position *vis-à-vis* her partners. Not only are leading German chemical combines extending their activities into neighbouring countries but, and this is more important, German technical and scientific help is diffusing to these countries. One wonders whether this is entirely agreeable to British and American scientific institutions.

Conclusions

The organising committee of the Symposium headed by Prof. Kramers, arranged the administrative and social side with noteworthy efficiency and taste. (This committee, it should be mentioned, was working in an entirely voluntary capacity and carried out the arrangements at very short notice.) Not only was the conference hall well chosen—tastefully decorated in old timber, which seems to be a favourite with the Dutch—but other facilities were there in abundance. On the social side it will be difficult to forget the bountiful hospitality extended by the Amsterdam municipality amongst the masterpieces of Van Gogh, and on a following evening the superb chamber music played at the Concertgebouw.

In view of the fact that the first Symposium on chemical engineering was held in Amsterdam in 1957, the next meeting of this kind is expected to take place in 1963. One very much hopes that it will again take place in Amsterdam—but the Germans should by then put in a strong claim for acting as host country.

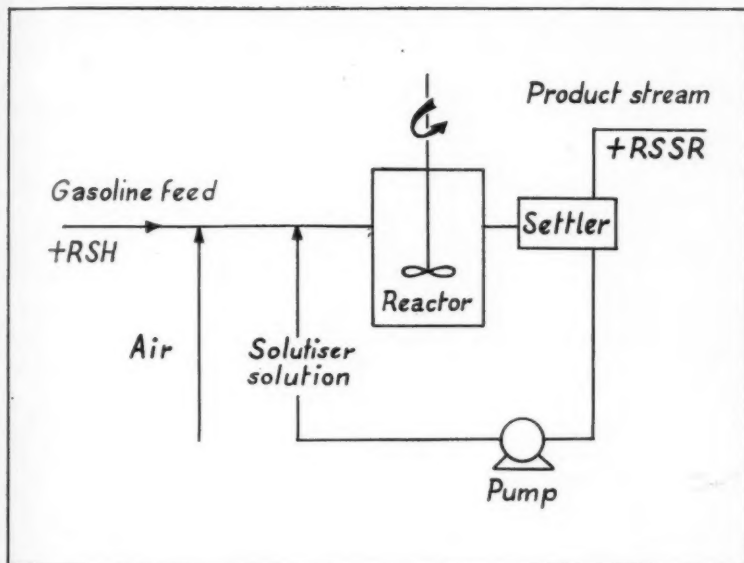


Fig. 2.

on physical and engineering factors. To complete the simulation a set of perturbations is added, corresponding as far as possible to the incidents of full-scale plant operation.

Significant impressions

The preceding synopses of some of the papers are sufficient evidence that the subjects dealt with were extremely varied and 'heterogeneous'. This is not surprising in view of the fact that the bulk of the membership at the Symposium came from so many different branches of chemical engineering. Be it noted, however, that all the authors and participants in

sium was made by Prof. Piret, American scientific attaché in Paris, who pointed out that this gathering had revealed a distinct European attitude and achievement in chemical engineering. The relatively young age of delegates and their enthusiasm for the subject was evidence that interesting developments were to be expected in reaction kinetics during the next decade. Many observers felt that the lead in this field may be slowly passing across the Atlantic from North America to Europe, more particularly Germany.

As an interesting comment, it has become quite noticeable that with the

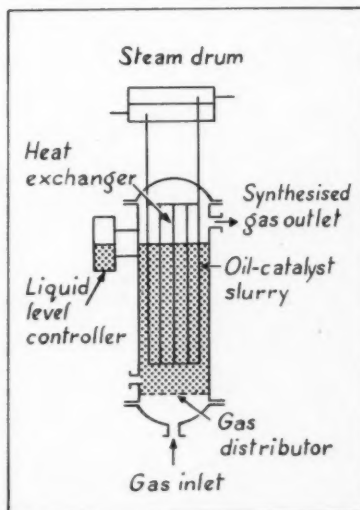


Fig. 3.

CPE Diary

JUNE 10 TO 26 British Trade Fair in the Coliseum, Columbus Circle, New York. Organised by the Federation of British Industries.

JUNE 12 TO JULY 14 Symposium on physical, chemical and biological methods in the study of high molecular weight carbohydrates to be held in the Edinburgh and East of Scotland College of Agriculture in Edinburgh. Details from the general secretary, The Chemical Society, Burlington House, London, W.1.

JUNE 12 TO 16 Annual meeting of the American Nuclear Society in Chicago, U.S.A. Details from the secretary, Mr. O. Du Temple, c/o John Crerar Library, 86 East Randolph Street, Chicago, Illinois.

JUNE 14 TO 16 Annual meeting of the Dechema Deutsche Gesellschaft für chemisches Apparatewesen eV. and the 26th meeting of the **European Federation of Chemical Engineering** and the eighth meeting of the **European Federation of Corrosion** in the Palmgarten at Frankfurt. Details from the Dechema, Frankfurt (Main), Postfach.

JUNE 15 TO 29 International Nuclear Congress and Exhibition on Electronics and Atomic Energy in Rome. Organised by the Italian National Committee for Nuclear Research. Details from secretariat, Rassegna Elettronica, Nucleare e della Cinematografia, Via della Scrofa 14, Rome, Italy.

JUNE 20 TO 30 Laboratory Apparatus and Materials Exhibition at the Royal Horticultural Society's New Hall, London, S.W.1. Organised by U.T.P. Exhibitions Ltd. Details from J. Wheaton, 6 Mercier Road, London, S.W.15.

JUNE 26 TO 27 Annual meeting of the American Society for Testing Materials at the Chalfonte-Haddon Hall, Atlantic City. Details from the society, 1919 Race Street, Philadelphia 3, Pa., U.S.A.

JUNE 27 TO JULY 1 Gordon research conference on nuclear chemistry at Colby Junior College, New London, New Hampshire, U.S.A. Organised

by the American Association for the Advancement of Science. Information from Dr. W. C. Parks, director, Department of Chemistry, University of Rhode Island, Kingston, R.I., U.S.A.

JUNE 27 TO JULY 8 International course, in French, on nuclear energy, designed for senior engineers and technical industrialists, at the Centre d'Etudes Nucleaires at Saclay, France. Details from O.E.E.C. European Nuclear Energy Agency, 38 Boulevard Suchet, Paris 16.

JULY 4 TO 5 International Congress on Catalysis in Paris at Maison de la Chimie. Organised jointly by International Congress on Catalysis Inc., a French national committee which acts as host, and the International Union of Pure and Applied Chemistry. Information from Dr. H. Heinemann, c/o M. W. Kellogg Co., 711 Third Avenue, New York, N.Y., U.S.A.

JULY 4 TO 7 2nd annual conference of the British Computer Society in the Sun Pavilion, Harrogate.

JULY 4 TO 8 Gordon research conference on chemistry and physics of isotopes at the New Hampton School, New Hampton, New Hampshire, U.S.A. Organised by the American Association for the Advancement of Science. Information from Dr. W. C. Parks, director, Department of Chemistry, University of Rhode Island, U.S.A.

JULY 4 TO 15 International course, in English, on nuclear energy designed for senior engineers and technical industrialists at A.E.R.E., Harwell. Information from O.E.E.C., European Nuclear Energy Agency, 38 Boulevard Suchet, Paris 16.

JULY 7 TO 13 Electronics and Instruments Exhibition and Convention at the Manchester College of Science and Technology. Details from W. Birtwistle, 78 Shaw Road, Rochdale.

JULY 18 TO 22 Nuclear Technology Survey at Berkeley, U.S.A. Enrolment limited to persons with **executive or managerial responsibility in industry**. Programme from the

Professor of Chemistry, Lawrence radiation laboratory, University of California, Berkeley, Calif., U.S.A.

JULY 18 TO 26 Tercentenary celebrations of the Royal Society. Ceremony in the Royal Albert Hall on July 19. Other activities include lectures and a conversazione at Burlington House. Information from Mr. M. Whitchurch, press officer, tercentenary celebrations, I.C.I. Ltd., Millbank, London, S.W.1.

JULY 18 TO 29 Summer programme on the dynamic behaviour of processes and equipment, especially where feedback control is utilised, at the Massachusetts Institute of Technology. Details from the Department of Chemical Engineering, M.I.T., Cambridge 39, Mass., U.S.A.

JULY 25 Lecture by Prof. Linus Pauling on the structure of metals and intermetallic compounds at the Royal Institution, Albemarle Street, London, W.1, at 6.30 p.m. Organised by the metal physics committee of the Institute of Metals. Visitors welcome, no tickets required.

JULY 25 TO AUGUST 6 General Assembly of the International Union of Geodesy and Geophysics including symposium on **atmospheric chemistry and radioactivity** and symposium on **dynamical processes in the atmosphere**, to be held in Helsinki, Finland. Information from the organising committee for the assembly, c/o Institut Geodesique, Boulevardi 40, Helsinki, Finland.

JULY TO AUGUST Symposium on technical and industrial applications of nuclear energy, including reactor technology and the economics of nuclear energy. To take place in Brazil. Organised by the Inter-American Nuclear Energy Commission. Details from the executive secretary of the commission, c/o P.A.U., Washington 6, D.C., U.S.A.

AUGUST 1 TO 12 International summer course in plasma physics in Denmark. Information from the International Summer Course in Plasma Physics, Danish A.E.C., Research Establishment, Risø, Roskilde, Denmark.

WHAT'S NEW



Plant • Equipment • Materials • Processes

CPE reference numbers are appended to all items appearing in these pages to make it easy for readers to obtain quickly, and free of charge, full details of any equipment, machinery, materials, processes, etc., in which they are interested. Simply fill in the top postcard attached, giving the appropriate reference number(s), and post it.

Cumene hydroperoxide

Cumene hydroperoxide is offered in commercial quantities by the Distillers Co. Ltd., selling agents for British Hydrocarbon Chemicals Ltd. It is available either in 5-gal. polyethylene containers or in 45-gal. polyethylene-lined drums.

It can be used as a polymerisation initiator and catalyst. It is said to be superior to other organic peroxides in the polymerisation of vinyl-type monomers because of the wide temperature range over which it is effective (100 to $-10^{\circ}\text{C}.$). It can be used in either homogeneous or in two-phase polymerisation as a solution in organic solvent or as the sodium salt in aqueous solution.

Cumene hydroperoxide also finds application as a catalyst for curing polyester resins. **CPE 1505**

Cooling tower

A new cooling tower, known as the type 'D', is offered by Foster Wheeler Ltd.

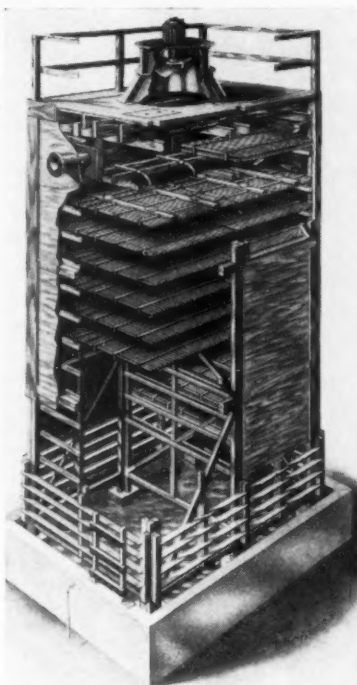
It is an induced-draught tower which will cool water quantities between 50 and 1,500 gal./min.

Constructed of treated timber and completely prefabricated, the unit is delivered to site in sections for erection by unskilled labour, including the fan, fan ring and motor, all sections being simply bolted together.

The water collecting basin forming the foundation can be constructed of concrete, steel or other suitable material. It is available in sizes from 6 x 6 ft. to 16 x 16 ft. **CPE 1506**

Lead shielding chambers

Lead shielding chambers for handling radioactive materials are offered in a packaged form by Associated Lead. Starting from minimum internal dimensions 26 in. wide, 24 in. high



Cooling tower.

and 26 in. deep, the buyer can merely specify the internal dimensions required. The complete chamber, including two sphere units for remote handling equipment and a standard lead window frame, is delivered in a case packed in sequence, to make assembly in unpacking a straightforward operation.

There are three standard chambers, all having 2-in.-thick lead walls, the same minimum internal dimensions, two sphere units for remote handling equipment and a standard lead window frame for observation. Type 'A' chamber is made up of 2-in.-thick solid wrought and precision machined

bricks to A.E.R.E. specification and design with chevron mating surfaces. Type 'B' chamber is made up of 2-in.-thick solid wrought and precision machined bricks with curved mating surfaces. Type 'C' chamber is made up of 2-in. extruded strip with serrated mating surfaces. By means of their unit construction, 'A' and 'B' type chambers can be increased in width by units of 8 in. and in height and depth by units of 4 in. The 'C' type chamber can be increased in all dimensions by units of 1 in. **CPE 1507**

Reflux divider

A reflux divider which is claimed to provide a simple answer to the problem of controlling reflux ratios for batch and continuous distillation plant is offered by Metal Propellers Ltd. It is said to be insensitive to rate of flow and give quickly reproducible settings over a range of ratios including zero and 100% reflux. The plant operator is able to select the correct sequence of reflux ratios and repeat these at will by setting the operating lever against the appropriate scale readings.

Flow through the divider is through two rectangular ports which are controlled by one lever.

The total width of these ports is always constant, but the relative width is variable, thus giving a continuously variable reflux ratio over a wide range. The selected ratio is maintained, irrespective of flow and working pressure, within the operating range of the particular size of reflux divider. **CPE 1508**

Temperature recorder

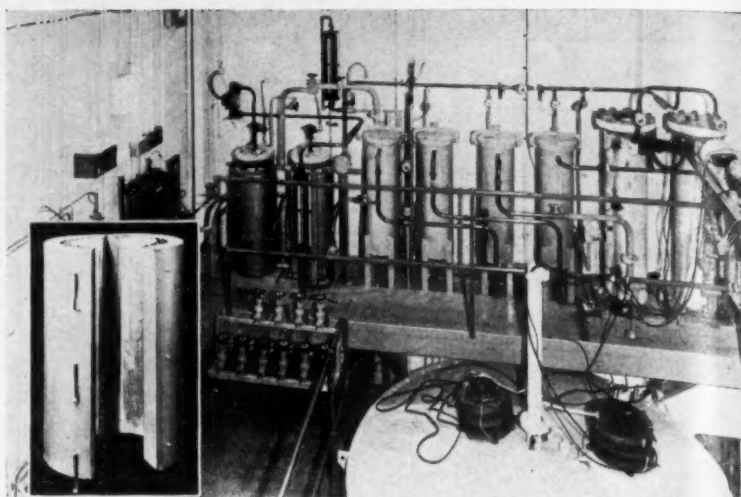
The *Minican* temperature recorder can be used under unusual conditions, in cooking vessels and in sterilising processes.

The pocket-size instrument consists of a chart 40-mm. wide mounted on a drum which revolves at a constant rate, permitting a pen to trace a temperature record for 2, 12 or 24 hr. The pen is moved by the expansion and contraction of the liquid in a stainless-steel bulb. The drum is operated by a built-in mechanical clock.

In use, both chart and pen are enclosed in a chromium-plated, liquid- and gas-proof brass cylinder, capable of standing up to high cooking pressures, so that the instrument may be enclosed in ovens or cold stores.

Accuracy of 0.5 mm. (0.02 in.) on the chart width is to be expected and the minimum range is 50° (F. or C.) across the chart. The makers are Cambridge Instrument Co. Ltd.

CPE 1509



Nitrogen purification towers at Hunterston.

Coal packaged boiler

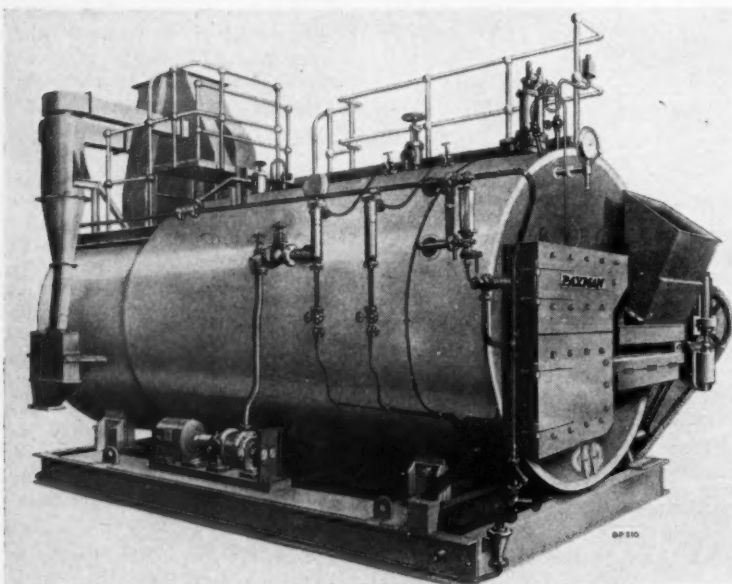
A coal packaged boiler was exhibited by Davey, Paxman & Co. Ltd. at the Fuel and Power Efficiency Exhibition. The *CoalPak* is a self-contained unit, mounted on its own base, and can be delivered ready for operation requiring connections only to the main services.

The boiler is available in eight sizes with overall lengths from 17 ft. 6 in. to 21 ft. 3 in. and outputs from 2,000 to 8,000 lb./hr. of steam from and at 212°F. It is mechanically fired by Hodgkinson low ram coking stoker.

At maximum firing loads the stoker is running continuously; for partial loads an automatic turn-down range is provided giving progressively shorter periods of firing as the load demand is lowered. Automatic coal feeding to the stoker hopper can be added by the provision of a suitable elevator and, if required, the coal feed to the stoker hopper can also be automatically regulated.

To meet the requirements of the Clean Air Act, a suitable dust collector is combined with the induced-draught fan, which is of the cyclone type and discharges into a hopper at the rear of the boiler.

CPE 1510



Coal packaged boiler.

Acidity control

The Paterson *Aquascope* is a portable instrument which makes use of indicators to determine the acidity in materials being processed.

It consists of a plastic moulding carrying a Perspex three-cell sampling unit arranged to permit the sample to be viewed through observation windows in front. The three compartments of the sampling unit are filled up to the level indicated by a black line with the liquid to be tested and the indicator solution is added to the central cell by the graduated pipette provided. After stirring, the colour produced is matched by inserting the appropriate colour slides in front of the two end cells which contain the liquid only. The nearest colour to that developed in the central cell is a measure of the pH of the sample.

The set of colour slides consists of nine permanent colour standards mounted in moulded slides of a shape convenient for manipulation, which are arranged in the right-hand compartment in two rows banked for easy access. The pH value is engraved on the top of each slide, and the appropriate indicator on the side.

The standard indicator supplied is brom thymol blue, which has a range from pH 6.0 to 7.6 in steps of 0.2. Should the colour produced be intermediate between that of two consecutive slides the pH can be expressed as an intermediate value, e.g. 6.9, 7.1, etc.

A selection of indicators and standard colour slides for testing samples of an acidity up to 4.4 or an alkalinity up to 10.0 is obtainable. **CPE 1511**

Heating jackets

The nitrogen purification plant of the ZENITH reactor, engineered by the G.E.C. atomic energy division, is designed to remove carbon monoxide, carbon dioxide and moisture from the coolant gas by passing it through a series of towers containing granular chemicals.

The first set of these towers seen in the photograph contains pellets of copper oxide. This is used to oxidise the CO in the coolant gas to CO₂ which is removed by another chemical in the next set of towers. For the copper oxide bed to react in this way it has to operate at a temperature of 400 to 450°C. and the pellets are most conveniently raised to this temperature by surrounding the towers with cylindrical heating jackets made by Isopad Ltd.

These units comprise the heating surface, thermal lagging and outer metal casing; the hinged construction allows simple and quick installation and assembly of the heater.

The bed temperature is controlled by a thermostat embedded in the bed of pellets. After a period the copper oxide beds are reduced to copper and it becomes necessary to 'regenerate' them. This entails oxidising the copper by passing oxygen over it at a similar elevated temperature. **CPE 1512**



LIQUID METER

Walker, Crossweller & Co. Ltd. are manufacturers of the Arkon meter which is reputed to measure almost any known liquid accurately and without interruption. The materials of construction can be altered to measure liquids ranging from chemicals and hot or cold water to syrups, oils, thinners and solvents. It is claimed that the viscosity of the liquid presents no obstacle, and fluids as different as petrol and grease can be measured with continuous and consistent efficiency.

The meters have a large capacity and can often be used in lines where a larger meter would seem necessary. Maximum line pressures of 150 p.s.i. can be recorded by all standard meters, while higher readings can be obtained from meters of increased size and capacity.

CPE 1513

Fill in and post the reply-paid card for details of any items in these pages, making sure to quote the correct 'CPE' reference number.

Differential pressure transmitter

A differential pressure transmitter used to convert a differential pressure to a proportional pneumatic output signal of 3 to 15 p.s.i.g. for the operation of remote flowrate indicators, recorders or controllers is offered by Fisher & Porter Ltd. It is a force-balance type with continuously adjustable damping, built in over-range protection and it is temperature compensated.

Its range is continuously adjustable from 0 to 20 in. to 0 to 200 in. water gauge and the zero is externally accessible. By inverting the range adjusting nut the range can be further increased to accommodate from 0 to 30 in. to 0 to 300 in. water gauge with equal facilities for adjustment. Accuracy is claimed to be 1% of full range.

It has a stainless-steel body of through-bolt construction with its working parts enclosed in a fibre-glass cover. No critical parts are touched by the process fluid. **CPE 1514**

Bellows joint

A new bellows joint has been added to the range of pipe suspension equipment of Vokes Genspring Ltd. The Genflex joints are used principally to absorb the expansion and contraction of pipe systems owing to temperature changes. The unit is compact, flexible and designed to absorb vibration and movement in all planes.

The standard bellows is produced from 18/8 cold-rolled stainless-steel sheet, welded into a tube with a longitudinal butt weld under accurately controlled conditions, the weld being the same material thickness as the adjacent metal. Convolutions are then formed on special machines. The joints are produced without any circumferential welds.

For special applications, they can be manufactured from Nimonic 75 for temperatures over 750°C., and Monel, Inconel and titanium where unusually corrosive conditions occur. Rectangular and other special bellows can be fabricated to large sizes. Diameters above 120 in. bore can be made to customer's requirements.

CPE 1515

Multi-purpose valve

A unit which provides a standard globe valve with a choice of trims to meet the particular needs of specific applications is the Bestobell Two-Five-O offered by Bell's Asbestos & Engineering Ltd.

The valve is a multi-purpose bronze globe valve with working pressures of steam up to 250 p.s.i. and cold up to 400 p.s.i.

The valve is offered with six alternative trims, loose valves and seats, to cover the whole range of common factory service—steam, water, air and gas—at temperatures and pressures most frequently encountered for control and stop purposes. **CPE 1516**

Oil-seals

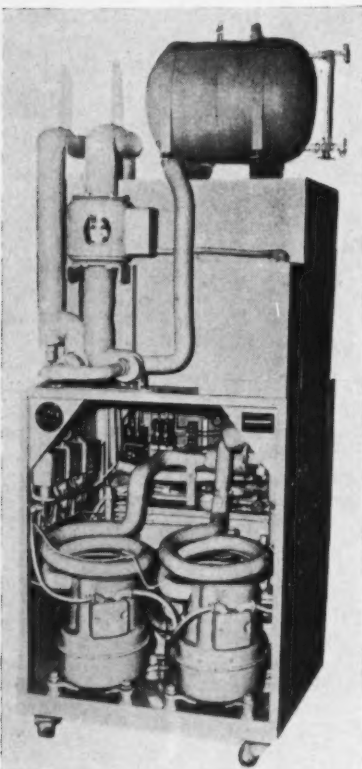
Precision-moulded oil-seals designed to reduce the risk of extrusion under pressure are offered by B.A.L. Ltd. The seals, made of Hycar nitrile rubber, are of one-piece construction.

The design and flared walls are claimed to provide a positive yet resilient fit on rods and cylinders

which ensures a seal at zero pressure and even distribution when under pressure.

Resistance to extrusion is achieved by using a fabric base which also reinforces the seal at the point at which stress and pressure are concentrated.

They are said to be particularly suitable for use on piston heads or the glands of reciprocating rods. *Hycar* nitrile rubber is made by British Geon Ltd. **CPE 1517**



View of the back of the brine chiller.

Portable refrigeration assembly

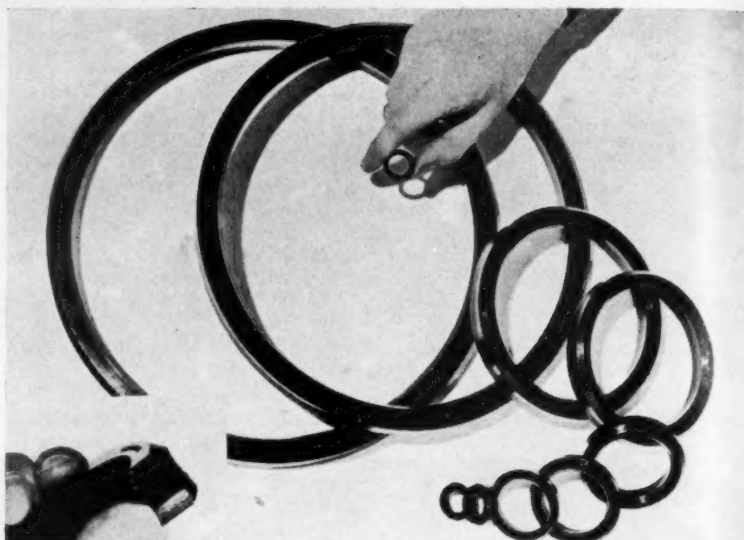
A one-piece, portable, packaged, mechanical refrigeration assembly which provides low-temperature mediums to any enclosure through heat exchangers that can be connected to the required flow lines is the *Kold-Pak* refrigeration equipment made by Tenney Engineering Inc. The equipment is either of the direct expansion type, with freon refrigerants passing through a user's heat exchanger, or the brine chiller type with a liquid acting as a secondary cooling agent.

There are 12 standard models with the following capacity span.

At -40°F. brine temperature or -60°F. evaporator temperature, the capacity is from 3,240 to 34,000 B.Th.U./hr. At -100°F. brine temperature or -120°F. evaporator temperature, capacity ranges from 660 to 7,000 B.Th.U./hr.

The equipment is said to be suitable as a source for rapid temperature changes in testing and in production for adaptation to low-temperature baths, cold traps (vacuum equipment), quenching baths and metal treatment.

In atomic energy areas, refrigeration can be provided in a 'hot' zone without endangering personnel. Expendable liquid cooling agents can be disposed of as they become contaminated. **CPE 1518**



A selection of moulded oil-seals. The inset shows a cross-section of one of these, illustrating the fabric reinforcement at the point where stress and pressure are concentrated.

Pipe joint seal

By using *Threadseal* tape it is claimed that a pipe joint can be made without soiling the hands, the same sealing medium can be used for whatever liquid is being handled, and the joint can be broken down after several years without difficulty.

The tape is made from PTFE and supplied in spools loaded with 480 in. of $\frac{1}{2}$ -in.-wide tape. There is no wastage as it is only necessary to cut off a sufficient length of tape to wrap round a thread, allowing for an overlap of about $\frac{1}{2}$ in.

The makers, Crane Packing Ltd., say it can be used on threads formed in virtually any material in which thread connections are formed. As it is self-lubricating it eliminates galling

and seizing and connections can be drawn up to a greater degree of tightness than with conventional sealing substances.

It can be used to seal against pressures of many thousands of p.s.i. and temperatures of -200 to +500°F. **CPE 1519**

Cryogenic equipment

The characteristics of the cycle employed in the refrigeration units of Philips Cryogenic Equipment are such that a relatively high torque is necessary to start the engine, this falling off as the special compressor picks up speed.

The makers have now developed a system whereby this high load on start-up is avoided. The basic principle of the system is that the compression ratio across the main piston is much reduced during the initial few revolutions of the crankshaft by the connection of a buffer vessel to the compression space in the cylinder. A valve in the line between the two is controlled by the oil pressure in the machine and remains open until the pressure generated by an integral pump driven from the crankshaft reaches its normal level. This valve then closes, the compression ratio rapidly reaches its operating value, and the machine starts to run at full speed.

The equipment is marketed by Research & Control Instruments Ltd. **CPE 1520**



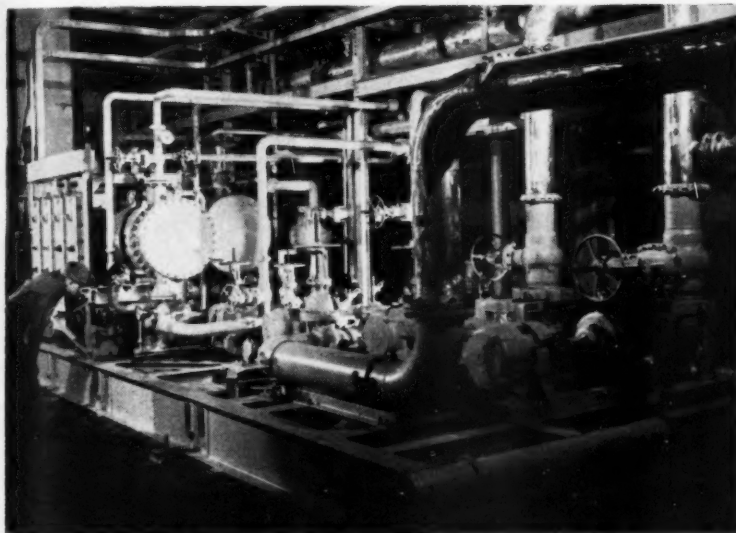
NUCLEAR NOTES

Euratom-U.K.A.E.A. co-operation

Under the agreement made last year for co-operation between the U.K.A.E.A. and Euratom, various meetings of specialists from the two sides are being held from time to time. One such meeting took place at the headquarters of the Authority's development and engineering group at

Risley where 50 experts from the Euratom countries, led by Mr. P. Caprioglio of the Euratom staff, discussed the U.K. advanced gas-cooled reactor system and the complementary European work.

Lectures were given by British, French, German and Italian speakers.



SKID-MOUNTED NUCLEAR POWER

The heat exchanger of the skid-mounted nuclear power reactor built to power a remote U.S. Army base on the Greenland ice-cap. The heat exchanger skid re-heats water from the condenser of the reactor prior to re-entry of the water into the plant's steam generator. The reactor system features an air and ethylene-glycol condenser cooling system, made necessary by the frigid temperatures. Power plant was designed by ALCO Products Inc.

I.A.E.A. current session

The International Atomic Energy Agency (I.A.E.A.) will send 20 nuclear experts in 1960 to Afghanistan, Austria, Brazil, Iran, Iraq, Korea, Philippines, Turkey, Venezuela and Yugoslavia. Austria, Iraq, Philippines and Yugoslavia will receive some equipment at a total value of \$47,200 needed in connection with the work of the experts.

This decision was taken by the board of governors at its current session, when it allocated 60% of the Agency's own technical assistance funds for the current year. The allocation was made on the basis of

requests for assistance under the 1960 programme received by the end of January, the remainder of the funds being left for requests received by the end of May. In addition, the Agency implements an assistance programme under the United Nations' expanded programme of technical assistance.

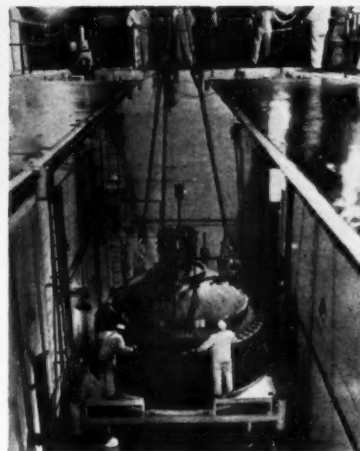
State of knowledge of uranium deposits

A review of the present state of knowledge about the exploration and evaluation of uranium and thorium deposits has just been published by the International Atomic Energy Agency. Entitled 'Surveying and

Evaluating Radioactive Deposits', the paper is the first in a series of reviews of recent scientific developments in different fields of the peaceful utilisation of nuclear energy.

It deals with the practical problems of the exploration and evaluation of radioactive deposits. It does not deal with prospecting except in so far as prospectors have to evaluate their discoveries in a preliminary way and certain techniques are common to both prospecting and testing of discoveries. Evaluation comprises not only the estimation of size, content and value of a deposit but also the methods of testing that provide data for such estimates. Exploration and evaluation are discussed with reference to the techniques which are peculiar to deposits of uranium and thorium, such as radiometric assays and surveys.

The review is based mainly on Canadian and U.S. experience.



DRESDEN POWER PLANT

One of the last steps before producing steam for the first production of nuclear electricity at the Commonwealth Edison Dresden station came when technicians placed the 50-ton head on the reactor vessel.

The massive top was bolted to the vessel body by 56 five-in.-diam. bolts.

G.E.C. designed and built the 180,000-kw. plant, the largest privately-financed, all-nuclear plant in the world, for Commonwealth Edison and the co-sponsoring Nuclear Power Group Inc.

The Dresden plant, 50 miles southwest of Chicago, is expected to be in full operation this summer. At full power, it will supply the needs of a city of 200,000 people.

Lignosulphonates from Waste Sulphite

By A. E. Williams, Ph.D., F.C.S.

In last month's issue we drew attention to novel utilisations for waste sulphite developed in Canada, the Atomic Suspension Technique for generating waste heat and the prevention of frost-heaving in soils by spraying with sulphite liquors. Another utilisation, also from Canada, is the production of lignosulphonates from waste sulphite liquors described in this article.

WASTE sulphite liquor—a by-product in the production of pulp and paper from wood—is often regarded as a useless material, sometimes presenting problems in its disposal. Much research, particularly in the timber-growing countries, has been directed towards converting the waste product into useful material. So successful has this been that North American authorities now consider that the by-product may become more profitable than the primary materials pulp and paper.

The basic principle on which progress in utilising the waste liquor relies is the fact that lignosulphonates can be produced from the lignin in the liquor. Lignin, comprising from 35 to 40% of the dry weight of wood, is separated from cellulose by sulphonat-

ing with a calcium compound to yield calcium lignosulphonate. This is then converted to either sodium- or ammonium-base liquor prior to concentration, firstly in evaporators and finally in spray driers to produce a solid product. Lignosulphonates are applied in industry in many ways. In foundries as core binders and for mould washing. Road constructors use them for dust-laying, highway stabilisation, treatment of frost-heaves and for impermeabilisation of silts and clays in road foundations. Cement and concrete industries employ them for reduction of moisture in cement rock slurries, as cement clinker grinding aids and as concrete plasticisers. The coal industry uses them in mine dust-laying, for briquetting and pelleting coal dust to make usable fuel.

The potential market has become so promising that two plants have been put on production in North America during 1959, one in Bellingham, Washington, U.S.A., and the one in Quebec, Canada, which is considered here. The latter is that of Lignosol Chemicals Ltd., a jointly-owned subsidiary of Anglo-Canadian Pulp & Paper Mills Ltd. and Anglo-Newfoundland Development Co. Ltd.

Spray drying

As the end-product in the process best performs its duties when in the form of fine spherical particles, the method of drying the material recovered from the sulphite liquor is spray drying. In 1952, the newly-formed Lignosol Co. was centred around two spray driers giving a combined production of 30 tons/day. In 1958, as the demand for lignosulphonates increased, the company initiated a \$1-million expansion programme, in which the major unit was a 100,000-lb./day spray drier, engineered and installed by Bowen Engineering Inc.

The new large-capacity spray drier went on stream in March 1959, boosting potential capacity well above the present supply of sulphite liquor in a concentrated form. An additional evaporator is being erected at the plant to increase the supply of concentrated liquor, and this will enable the three spray driers to produce 80 tons/day of the finished product. The new 50-ton giant drier, one of only two of its size in the drying of lignosulphonates in the world, was chosen by Lignosol. Two types of driers were initially considered, one with a conical base chamber, the other with a flat base; the latter was eventually chosen because of its several advantages.

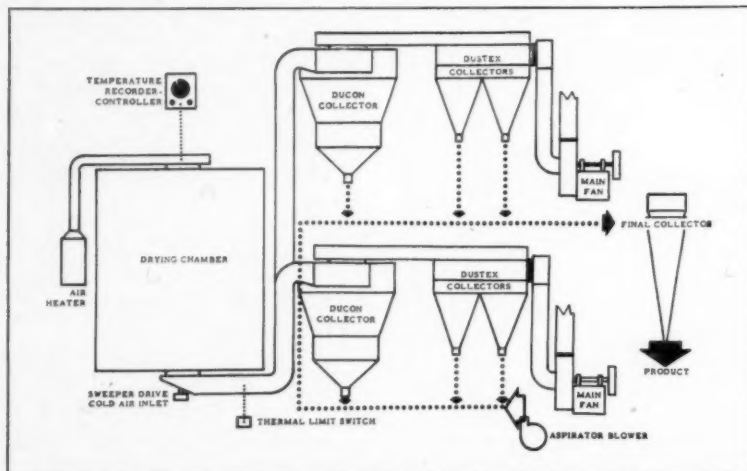


Fig. 1. Simplified flow sheet of spray-drying process at Lignosol Chemicals Ltd. This simplified drawing is taken directly from original engineering drawing provided Lignosol by Bowen Engineering Inc., the firm which engineered, fabricated and installed the giant drier.

Outline of process

All spent sulphite liquor, after chemical treatment, is received at the Lignosol plant from the adjacent Anglo-Canadian mill 100 yd. away by pipeline, and from the liquor a 50 to 55% slurry is prepared which forms the feed for the spray driers. The essential features of any spray drier are that the material to be dried must be presented to the heat source in the form of a very fine spray or fog-like mist. Being finely divided, the moisture in the product is instantly evaporated as it contacts the heat; thus a product is at the maximum temperature for only a second or so before it reaches the cooling zone of the drying

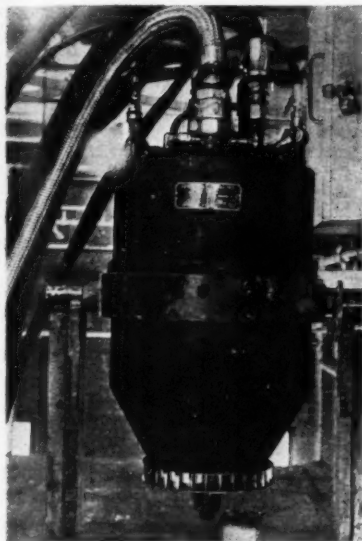


Fig. 3. The 'Bowen' patented high-speed spray machine with 30-vane atomiser wheel which runs at 12,000 r.p.m. Power is derived from a frequency converter driven by a 40-h.p. motor.

Bakeable valve

Many of the U.K.A.E.A.'s research and production laboratories have installed considerable numbers of vacuum valves using expendable metal or non-metallic seals.

However, existing types of metal seals have a very short operational life involving a lot of maintenance, while non-metallic seals cannot be used at high temperatures.

To get round both these failings, Harwell engineers and metallurgists have developed a sealing system which provides a new metal seal at each closure to give an efficient vacuum over a much longer life.

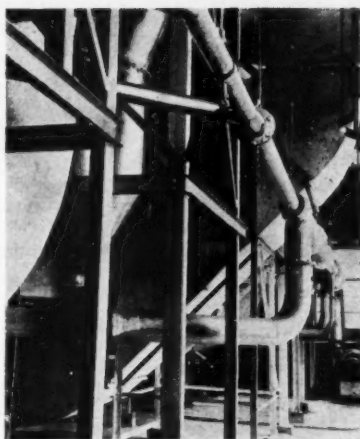


Fig. 2. Two 'Dustex' collectors on the left, main fan ducting in centre, aspirator blower in right rear, cooling and collecting pipelines overhead and foreground.

chamber. Collection of the spherical particles from the drier occurs in two Ducon cyclone collectors followed in series by two *Dustex* collectors (Figs. 1 and 2) and powder from both sources is conveyed to a central point by the use of an aspirator blower which conveys the material to a final collector unit, serving as a feed hopper to the packing department.

The high-speed spray machine which atomises the slurry in the drying chamber consists of a high-frequency motor with a patented 30-vaned atomiser wheel of stainless steel mounted on the motor shaft, which spins at approximately 12,000 r.p.m. (Fig. 3). Power is supplied to this spray machine by a frequency converter driven by a 40-h.p. motor. Speed may be decreased or increased by changing the drive sheaves on the converter, thus changing the current frequency going to the motor. This

It consists of a deformable metal valve seat washer which, by plastic flow, forms a seal through pressure from a flange on the nose portion of the valve.

When the nose portion is withdrawn, its flange forces the washer to a position similar to its original position in the valve seat.

Work-hardening may occur after repeated use, but this can be relieved simply by baking the entire valve to anneal the washer—giving additional useful life to the valve without any need to dismantle it.

Materials from which the deformable washer can be made include gold,

was considered to be a substantial improvement on the use of gears or belt drive for obtaining the high speed.

The heating medium used in the drier is hot air, provided by a compact space-saving burner incorporating steam atomisation of oil. This type of burner was preferred after considering other heater designs in that it permits faster start-up from cold than is possible with conventional furnaces, so offering a substantial fuel saving. The burner uses what is known in Canada as Bunker C oil.

Drying

The 50 to 55% total solids feed enters the drying chamber through the spray machine at a rate of from 70 to 140 lb./min., depending on the drying conditions required to meet specifications for particular grades of product. Output moisture content ranges between 2 and 4%.

Residence time of product in the chamber is a matter of only a few seconds, from slurry to dried product of predetermined moisture content. A constant inlet temperature, ranging from an average of 500°F. for most slurries to 400°F. for particularly heat-sensitive products, is maintained by a temperature controller which acts on the oil supply to the burner. Outlet temperatures range from 250 to 275°F. and are regulated by a similar controller acting on the feed supply. The controls are completely interlocked to ensure careful temperature and quality control. Cold air is introduced into the drying chamber at the bottom by a patented air-sweeper to shock-cool the particles. This shock-cooling and exact temperature control throughout is of prime importance, for excessive heat, or prolonged exposure to heat, could easily be deleterious to the products.

platinum, palladium, copper, aluminium, lead or alloys of these having suitable ductility. The valve spindle and body would normally be made of stainless steel.

Graphite temperature measurement

Following the Wigner release accident at Windscale, a new instrumentation panel containing seven strip chart recorders has been installed on BEPO research reactor at A.E.R.E., Harwell. These multiple-pen instruments give a permanent record of graphite temperatures and were supplied by Honeywell Controls Ltd.

New Books

Fast-Neutron Spectroscopy. By B.V. Rybakov and V. A. Sidorov, Consultants Bureau, New York. Chapman & Hall, London, 1960. Pp. 121. \$17.50 net.

Once one has recovered from the shock of the cost of this volume, one can feel little except enthusiasm for it. The translation is excellent, and has brought out clearly the masterful exposition by the authors of some fascinating features of Soviet research in a very important field. This book is based on work on what may be the finest existing spectrometer, while the time-of-flight methods here described refer largely to the authors' own experiments and results.

Electronic techniques in the 0.3 to 30-MeV energy range are described in detail, emphasis being on time-of-flight method, which is new in this particular energy range. The contents include chapters on recoil-nucleus method, proportional and scintillation counter telescopes, nuclear reaction method, pulsed neutron sources, neutron detectors, time analyser, basic features of time-of-flight spectrometers and an energy/time-of-flight table.

The text is profusely illustrated by line diagrams which, happily, are large enough to be clearly legible. It is a pity, however, that so many of these are incompletely labelled. To take one example, the legend under Fig. 20 refers to labels *a* to *g*, which do not appear on the diagram. This fault is all too frequent. There are also several errors such as 'if the nuclei . . . is introduced . . .' Each chapter has a useful list of references to relevant literature. This book is certainly a necessity for anyone working in, or interested in, this particular field of nuclear physics.

F. R. PAULSEN

£ s d

CHEMICAL PLANT COSTS

Cost indices for the month of April 1960 are as follows:

Plant Construction Index: 180.2

Equipment Cost Index: 169.0

(June 1949 = 100)

£ s d

The Radiochemistry of Cadmium. By J. R. Devoe. Pp. vi + 57. \$0.57.

The Radiochemistry of Arsenic. By H. C. Beard. Pp. vi + 27. \$0.50.

The Radiochemistry of Francium. By E. K. Hyde. Pp. vi + 34. \$0.50.

United States Atomic Energy Commission Monographs. Available from the Office of Technical Services, Department of Commerce, Washington 25, D.C., U.S.A.

These are the first of a series of monographs on the radiochemistry of the elements, sponsored by the Subcommittee on Radiochemistry of the Committee on Nuclear Science of the U.S. Academy of Sciences-National Research Council. Each publication has been written by an expert on the radiochemistry of the individual element, following a standard format. It is expected that about 50 more of these volumes will be issued within the next few months.

The publication of the series of monographs is particularly desirable, as many radiochemical procedures have been recorded in diverse atomic energy reports and not in scientific journals. The new volumes should prove of value not only to radiochemists but also to a number of scientists interested in such fields as inorganic and analytical chemistry, nuclear physics, geochemistry, nuclear engineering, electronics, biology and medicine. It is planned to revise the monographs periodically to keep them up to date, and the reader is encouraged to call to the attention of the authors material which might be included in revised versions.

In each of the three first volumes features of the chemistry of the individual element of chief interest to radiochemists are reviewed. Tables of the isotopes of the elements are presented, and brief details as to the mode of preparation of the radioisotopes are included. Pertinent counting techniques are discussed. In each monograph major attention is given to the radiochemical separation and purification of the radioisotopes, and detailed accounts are given of many radiochemical procedures. The majority of relevant references are listed.

The volume on the radiochemistry of cadmium includes useful general

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data on the solubility and stable weighing forms of precipitates, stability constants of complexes, solvent extraction, ion exchange and gamma-spectra. The monograph on arsenic also gives much valuable practical and general information, and the author suggests a radiochemical procedure in the light of methods evaluated. As this radiochemical procedure and many of the others described involve distillation and hypophosphite reduction, it is felt that the data due to Smales and Pate (*Analyt. Chem.*, 1952, **24**, 717) on arsenic recovery in these steps could have been quoted with advantage. Another point, which might have been mentioned, is that the dissolution of some analytical samples, e.g. silicon, in sodium hydroxide solution with an excess of hydrogen peroxide, may be disadvantageous and lead to losses of radioarsenic (see Smales, Mapper, Wood and Salmon, A.E.R.E. C.R. 2254 (1957)). It is felt also that the gamma-spectrometry of radioarsenic should have been remarked upon.

The monograph on the radiochemistry of francium is interesting, as francium is likely to remain the most unstable of the first 100 elements. Since francium cannot be obtained in other than ultramicro-quantities, it follows that the practical chemistry of the element is identical with its radiochemistry.

The Subcommittee on Radiochemistry of the U.S. National Academy of Sciences-National Research Council are to be congratulated on sponsoring the new series. The first three volumes are mines of useful and significant information, and many scientists must look forward with enthusiasm to the issue of the further monographs.

D. F. C. MORRIS

Nuclear Reactor Optimization.

By P. H. Margen. Temple Press, 1960. Pp. 81. 12s. 6d. net.

This little volume shows the imprint of the master; it is lucid, well thought out and concise without being obscure. The author's theme is based on the concept that in any one type of

reactor there are more than 10 independent variables which can be changed without adversely affecting safety or power output. Obviously, only one combination can give the best results. Optimisation consists in finding this best set of conditions. Without such optimisation, about one million different designs would have to be studied.

By following the author's scheme, a designer and a physicist could achieve the optimum concept in a fairly short time, without the use of a computer. Mr. Margen illustrates his arguments by reference to a 250-Mw., high-temperature, gas-cooled reactor, moderated by heavy water, although the scheme would be equally applicable to any other heterogeneous system working at thermal energies. The first chapter deals with optimisation of a simple plant item, actually a copper electrical conductor. Then comes a discussion on reactors, covering generation costs, physics, core and reflector. The type of core having been selected, the next step is realisation of the variables and practical limitations. The advantages of core enrichment are brought out, and then heat exchange is studied, together with thermodynamics.

The appendices deal with cost formulae and data, a worked example on design sequence, and tabulated optimisation calculations. This book is both helpful and stimulating, and the only drawback is the cost, which is still high for such a slender volume.

F. R. PAULSEN

Atomic Energy in the Soviet Union. By Arnold Kramish. Stanford, California: Stanford University Press. London: Oxford University Press, 1960. Pp. 232 + x with notes, bibliography and index. 27s. 6d. (in U.K. only).

The Soviet Government has not so far issued an account of the early development of atomic energy in the U.S.S.R. Arnold Kramish of the Rand Corporation has produced a very readable book on this subject which can be recommended for general reading to those interested in the development of nuclear physics and atomic energy. A wide variety of sources have been used to produce a chronological story beginning with the early Russian work on radioactivity and nuclear physics, and ending with a short account of the Soviet interest in thermonuclear reactions. While the book is not a serious technical work,

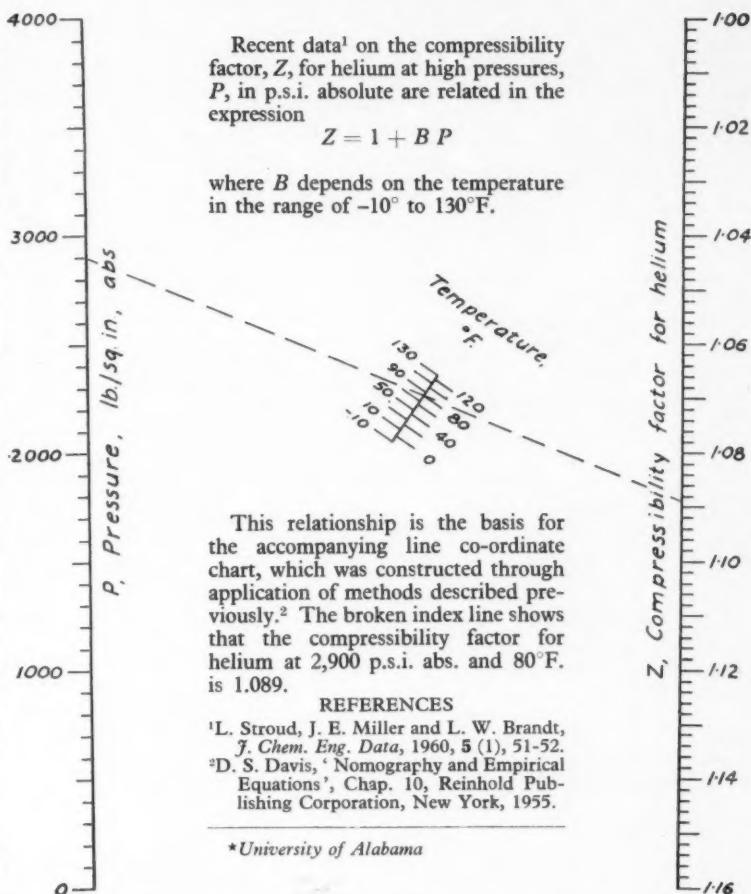
Compressibility Factors for Helium at High Pressures

By Prof. D. S. Davis*

Recent data¹ on the compressibility factor, Z , for helium at high pressures, P , in p.s.i. absolute are related in the expression

$$Z = 1 + B P$$

where B depends on the temperature in the range of -10° to 130°F .



This relationship is the basis for the accompanying line co-ordinate chart, which was constructed through application of methods described previously.² The broken index line shows that the compressibility factor for helium at 2,900 p.s.i. abs. and 80°F . is 1.089.

REFERENCES

- ¹L. Stroud, J. E. Miller and L. W. Brandt, *J. Chem. Eng. Data*, 1960, **5** (1), 51-52.
- ²D. S. Davis, 'Nomography and Empirical Equations', Chap. 10, Reinhold Publishing Corporation, New York, 1955.

*University of Alabama

In 'Pressure Drops Across Wet Drained Columns' on page 113 of the March 1960 issue the symbols Δp_w and Δp_d in the chart should be interchanged.

the arguments are developed logically and appear to be politically unbiased. Due to lack of evidence there are gaps in the story which can be filled only by intelligent speculation. This is particularly true of the sections dealing with the relationships between the government and the scientists and the search for the reasons underlying the major Soviet decisions in the field of atomic energy.

The author is not concerned with the technology of the development of atomic energy or in telling a story of scientific progress, but rather in tracing the growth of research institutes and the fortunes of leading scientists and

in recording the race to build the biggest particle accelerators and to gain international prestige by spending large sums of money on research.

In writing this book the author has set himself a difficult task. The book appears, however, to have been competently produced. References are given to the main sources of information and a bibliography on Soviet scientific and technical developments is provided for further reading. A chronological summary of the main events discussed in the book might have helped in giving the reader a more definite lasting impression of the subject.

S. F. PUGH

Orders and Contracts

Primary separation

The Mitchell Construction Co. Ltd. have been awarded a contract for the construction of a second primary separation plant at the U.K.A.E.A. Windscale works, Cumberland. The contract covers the main civil engineering and building works for the separation plant, which will service fuel elements received from the new civil atomic power stations.

The contract includes the main building, which is of mass concrete construction and of considerable complexity, involving the use of special concreting techniques, an associated office block, sub-stations and other ancillary structures.

Work will commence immediately and will take about three years to complete.

Magnesite ore preparation

Edgar Allen & Co. and one of its subsidiary companies, Buell Ltd., have obtained orders to the total value of £160,000.

The Edgar Allen engineering division will supply raw magnesite preparation equipment through the International Construction Co. Ltd., for installation at Salem, India.

The orders received by Buell Ltd. are for a vertical turbo dryer for Watts, Blake, Bearne & Co. Ltd., china clay manufacturers, capable of drying china clay from filter presses, and a vertical turbo dryer for Spillers Ltd. for the drying and cooling of maize flakes.

Laboratory extension

Work began recently on an extension to the Longbenton research laboratories of Thomas Hedley & Co. Ltd. at Newcastle upon Tyne.

The new accommodation will consist of offices, large and small laboratories and an extension to the existing library.

Construction is being carried out by Ralph Bowey & Son Ltd. at a cost of nearly £40,000. The building is to be completed by October and it is anticipated that all equipment will be moved in and the facilities in full operation before the end of this year.

Mobile linear accelerator

A development contract for a mobile linear accelerator designed to take X-ray photographs of the welds in the pressure vessel of a nuclear power

station has been placed by U.K.A.E.A. with Mullard Equipment Ltd. The machine is expected to be operational towards the end of the year.

The contract results from investigations, commissioned by the Authority, into the suitability of such machines for making radiographic inspections of welds in thick materials.

The accelerator will have an energy rating of 4.3 million electron volts and will give an X-ray output of over 600 roentgens/min. in air at 1 m. focus film distance. Because of its high energy rating it will make possible faster radiographs through greater thicknesses of material than is practicable with conventional X-ray or isotope sources.

The instrument is designed for use on site during the building of a nuclear power station with a minimum disturbance to the constructional work.

Polyethylene plant

Scientific Design Co. Inc. has been awarded a contract to engineer, design, and construct a polyethylene plant for Foster Grant Co. Inc., the first in the U.S. to utilise the *Agfo* process.

This process, developed by Imhico A.G. of Switzerland, is a continuous, high-pressure polymerisation process for producing polyethylene. It is highly instrumented, and by rigid control of polymerisation consistently

reproducible products can be made over a broad range of density, melt index and other relevant properties.

PVC production

P.G. Engineering Ltd. has been entrusted by Distillers Co. Ltd. with the engineering, design and procurement of a monomer plant and external services as a major stage in a project for the production of PVC.

Water treatment

William Boby & Co. Ltd. have been awarded a contract, valued at £2,170, for a small deaerator for the Metropolitan Water Board, and a contract valued at £3,500 for a demineralisation plant by the U.K.A.E.A. at Harwell.

Combustion gas turbo-compressors

The Richardson Westgarth Group received an order from one of the major oil companies for eight combustion gas turbo-compressors for overseas installation.

Steel strip lines

The Head Wrightson Machine Co. Ltd. have been awarded orders by Richard Thomas & Baldwins Ltd. for five steel strip cut-up lines for their new Spencer works. These lines, which will include a number of new features, are expected to be in full production by October 1961.

Colville's Ltd. placed an order with the same company for two steel strip lines to deal with hot-rolled material.

The value of the two orders is about £2.5 million.

Industrial Publications

Products and processes. A new, revised edition of 'Products Available from Union Carbide International Co.' has been published. It contains a complete list of the products and processes available from the company and the company's affiliated overseas companies and a list of distributors, indicating the products each has available.

Overhead materials handling systems. A handbook (Bulletin 23), published by British MonoRail Ltd., describes the *MonoRail* overhead materials handling system and shows the ways in which the system is applied to cater for different industrial handling requirements.

Aluminium alloys. A guide to the selection of aluminium alloys for virtually any product that can be made of the light, corrosion-resistant

metal is contained in a new booklet offered by the metals division of Olin Mathieson Chemical Corporation. It describes the physical properties, fabrication characteristics and economic advantages of a wide variety of aluminium sheet, plate, rod, bar, extrusion and casting alloys. The various tempers, finishes and patterns available are discussed. Aluminium's yield and tensile strength, thermal conductivity and electrical conductivity are graphically compared with those of other materials.

Flow measurement. Condensed Bulletin M-1, published by the instrument division of Schutte & Koerting Co., pictures and briefly describes the division's line of precision instruments for measuring, indicating, recording and controlling the rate of flow of all types of fluids.

Company News

Furnace division

Head Wrightson Processes Ltd. have been supplying furnaces to the petroleum and chemical industries for a number of years and, to meet their expanding operations in this field, they have formed an industrial furnace division at their London offices.

The company operates additional special divisions to deal with their design and manufacturing interests in cooling equipment and water purification plant.

Fertiliser and chemical projects overseas

Chemical Works Projects Ltd. has been formed by P.G. Engineering Ltd., Humphreys & Glasgow Ltd., and Simon-Carves Ltd. to supplement their normal activities by undertaking contracts for large fertiliser and chemical projects overseas. The joint company, thereby, will be able to call on extensive resources.

The board will consist of Mr. J. P. V. Woolam, Mr. G. Gresle Farthing and Mr. R. W. Rutherford, who will be the first chairman, each company having an alternate director.

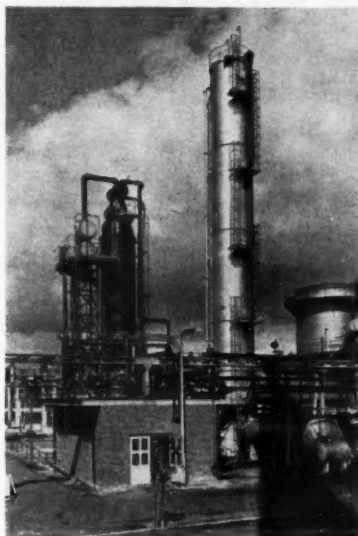
Detergent factory

Holland & Hannen and Cubitts (Great Britain) Ltd., are working on a £58,000 extension to the soap and detergents factory process building of Thomas Hedley & Co. Ltd. in Essex.

The extension, which will be completed by the end of the year, will have a structural steel frame on piled foundations and will be fire-insulated with reinforced concrete casing. Floor and roof slabs will be of flat reinforced concrete, and the external elevations will be brick-faced.

Benzole recovery

The construction of the benzole recovery and desulphurising plant at the Isle of Grain works of the South Eastern Gas Board was carried out by the Chemical Engineering Division of W. C. Holmes & Co. Ltd. The installation was completed in just under eight months. This plant, which has a daily capacity of 16 million cu. ft. of gas, operates on gas produced by the *Segas* process and is installed immediately after the dry purification plant. It operates on the well-known oil circulation system using vacuum distillation of the oil, which has the merit of maintaining the circulating oil in good condition more



Benzole refinery at Isle of Grain.

or less indefinitely. Another advantage of this type of plant is that it is extremely economical in steam.

An interesting feature is the inclusion of a naphthalene stripping section in the main vacuum still, this stripping section dealing with the oil from a naphthalene washer on another part of the plant. A further feature is the use of a steam jet vacuum pump as a stand-by to the main rotary vacuum pump.

Anglo-American enterprise

The Ralph M. Parsons Co. of Los Angeles and P. G. Engineering Ltd., have formed a new company, Parsons Powergas, with headquarters in London. Complete engineering and construction of petroleum, petrochemical and related plants will be performed by the new company.

Initially activities will be concentrated in the Commonwealth. The two firms will each appoint three men to a committee which will direct the new enterprise.

\$1 million to F.A.O.

A meeting of 41 representatives of the fertiliser industry has asked the Food and Agriculture Organisation of the United Nations to invite the industry to contribute \$1 million over two years for an international fertiliser programme under F.A.O.'s Freedom-from-Hunger campaign.

The objectives of the programme are to promote the efficient use of ferti-

lisers to increase food supplies in deficit areas and food supplies for animal production, to help develop national programme of fertiliser use and production, to assist in selecting methods of spreading information on fertiliser needs and use and to develop guidelines regarding fertilisers in foreign aid.

Work would include fertiliser tests on cultivator's fields, extensive demonstrations of fertilisers and development of soil-testing laboratories and services.

Merger

The boards of Albright & Wilson Ltd. and A. Boake, Roberts & Co. (Holding) Ltd. have reached agreement with a view to the merger of the two businesses.

Vinyl chloride polymers

I.C.I. Ltd. are considering proposals to extend the existing capacity for vinyl chloride polymers at their Hillhouse works by 10,000 tons, the extension to be in operation by the middle of 1961. Their present capacity for polymer manufacture is about 70,000 tons, compared with 55,000 tons in 1958. The increase to the present 70,000 tons has been achieved by the application of new manufacturing techniques on the existing plants. It is proposed to make an extension in PVC compounding capacity in parallel with the polymer extension.

The steady progress made in the use of PVC by industry, consumer durables and consumer goods is expected to continue. During the last three years advances have been made in developments new to the U.K., such as PVC piping for water supplies and drainage, gutterings, downpipes, soil pipes and similar constructional units for the building industry.

Zinc and lead extraction

Two companies of the Power-Gas group, Ashmore, Benson, Pease & Co. Africa (Pty.) Ltd. and P. G. Engineering Ltd. are to carry out an order valued at over £1 million for the Rhodesia Broken Hill Development Co. Ltd.

The order is for a zinc and lead furnace which will be installed at Broken Hill, Northern Rhodesia, and the contract includes the engineering, procurement and construction of the whole of the plant within the furnace area covering foundations, plant and equipment. Process design is being supplied by Imperial Smelting Processes Ltd. Completion is planned for the end of 1961.

International NU-SWIFT News

No. 2

HAVE YOU MET THESE PEOPLE?

19. IN KAINGAROA, NEW ZEALAND, largest man-made forest in the world, where pinus radiata trees grow faster than anywhere else on earth, no powered vehicle is allowed to enter unless equipped with an efficient fire extinguisher. N.Z. FOREST SERVICE has standardised on Nu-Swift extinguishers for all mobile equipment, including chain saws.

20. BRITISH EXPORTS of fire extinguishers are the highest in the world. NU-SWIFT exports are increasing in value continuously. Last year they amounted to NEARLY ONE-THIRD OF THE BRITISH TOTAL.

21. In arctic SPITZBERGEN (Norwegian: SVALBARD), divided by an invisible iron curtain, but where Norwegian and Russian workers fraternise, the NORWEGIAN COLLIERIES of STORE NORSKE SPITSBERGEN KULKOMPAGNI are protected by Nu-Swift.

22. THE ROYAL CANADIAN NAVY, which in 1947 standardised on the Nu-Swift Universal Water/CO₂ Extinguisher, has placed further orders.

23. HAILE SELASSIE, H.I.M. of ETHIOPIA, is reported to take a keen interest in fire extinguishers. Nu-Swift has been approved and the first consignments have been sent to ADDIS ABABA.

24. Narrow and winding alleyways in the ancient town of ST. GEORGE, first British settlement in now fashionable BERMUDA, sometimes prevent the Fire Brigade from getting close enough for effective fire fighting with hoses. Fire engines are therefore fitted with extinguishers to enable firemen to fight inaccessible fires with reliable Nu-Swift.

25. A.T.A.C., the MUNICIPAL TRAFFIC COMBINE of the City of ROME, and reputed to be the largest

surface passenger transport organisation in Europe, has decided, after tests extending over two years, to standardise on Nu-Swift.

26. YUGO-SLAVIAN STATE-OWNED CORPORATIONS are among the Nu-Swift customers situated on the edge of the East-West iron curtain.

27. In HELSINKI, capital of gallant little Finland, and almost within a taxi ride of Leningrad, the FINNISH POSTS AND TELEGRAPHS DEPARTMENT has decided to standardise on Nu-Swift Dry Powder extinguishers, especially suitable for use under SEVERE ARCTIC CONDITIONS, common each winter in Northern Finland. Substantial orders have already been placed.

28. GERMAN SHIPOWNERS also are now able to fit Nu-Swift in their ships, thanks to the approval of the SEE - BERUFGSGENOSSENSCHAFT in Hamburg.

29. At TULLN, Austria, in the presence of high-ranking Austrian fire-fighting officials, including BRAND-DIREKTOR DUFEK, Chief Fire Officer of Vienna, two Nu-Swift Dry Powder Extinguishers, Model 1604, bonded for 2 years for reliability tests, were recently discharged. They passed with flying colours and OBERBRAND-RAT SPEIL, the official responsible, signed the certificate of approval on the spot.

30. INDEPENDENT TELEVISION AUTHORITY, always in keen competition with the BBC, is determined that programme transmission shall never be interrupted through preventable causes. All its stations are now protected by Nu-Swift.

31. Reputedly being let at £25 a day or more, NEW LUXURY SUITES on the top floor of the DORCHESTER,

London's premier Park Lane Hotel, are protected by Nu-Swift.

32. The up-to-date works of ALFA ROMEO, the well-known motor-car manufacturers in NORTHERN ITALY, have recently been equipped with Nu-Swift. Crash tenders too have been fitted with fast and reliable Nu-Swift.

33. In NASSAU, BAHAMAS, El Dorado of YACHTING AND DEEP-SEA FISHING enthusiasts, many of the YACHTS are equipped with Nu-Swift.

34. H.R.H. PRINCESS BENEDIKTE, teenage daughter of the King of Denmark, recently launched a large train ferry named after her at ELSINORE, close to the legendary home of Hamlet. Required urgently by the DANISH STATE RAILWAYS to cope with increase in cross-water traffic, the ferry was equipped with Nu-Swift.

35. The Trustees of SHAKESPEARE'S birthplace in STRATFORD-ON-AVON, conscious of the dollar-earning value of the Bard's home, have protected the property with Nu-Swift. Last year the house was visited by over 200,000 sightseers.

36. H.M. THE KING OF SWEDEN has had Nu-Swift fitted in his cars and other vehicles. On board H.S.M.S. 'TRE KRONOR', Sweden's premier man-of-war, his Majesty is similarly protected. Three other reigning Sovereigns are also protected on board their yachts by Nu-Swift.

37. ESSO RESEARCH LTD. have equipped their new offices at ABINGDON, BERKS., ENGLAND, with Nu-Swift.

38. CHRISTCHURCH TRANSPORT BOARD in New Zealand, which puts TWO Nu-Swift extinguishers on each bus, specifies that extinguishers must be fitted before buses leave England, where they are made.

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CHEMICAL & PROCESS ENGINEERING, June 1960

Personal Paragraphs



J. G. Thorpe.



F. R. Brown.

★ **Mr. J. E. Monaghan** has been appointed manager of the rubber sales department at the London sales office of Midland Silicones Ltd. He replaces **Mr. P. A. J. Gate** who left the company to take up a position to start up a technical service department for Pentachemical Industries (Pty.) Ltd., a subsidiary of Engelhart Industries (Pty.) Ltd. in Durban, South Africa.

★ **Mr. B. Topley** has been elected deputy chairman of the board of directors of Albright & Wilson Ltd. He joined the company in 1936 and was appointed a director in 1944.

★ At the annual general meeting of the British Rubber and Plastics Belting Manufacturers' Association **Mr. D. D. Marshall** of the Greengate & Irwell Rubber Co. Ltd. was re-elected chairman for 1960-61.

★ **Mr. H. McGlyn** has been appointed quality control engineer at the Brighton factory of West Instruments Ltd.

★ **Mr. S. L. Waide**, director in charge of the chemicals division of Newton Chambers & Co. Ltd. since 1958, has been elected chairman of the British Disinfectant Manufacturers' Association and also chairman of the Association of Toilet Paper Manufacturers.

★ **Mr. J. F. Willsher** has been appointed to the board of directors of George Kent Ltd. Since early last year he has been general works manager.

★ **Mr. J. W. Plowman** has resigned his directorship of Dewrance & Co. Ltd. due to health reasons. **Mr. B. S. Bass**, previously sales manager, has been appointed in his place to the board as sales director.

★ **Mr. F. R. Brown** has been appointed manager of personnel, welfare and training of the Press group of companies. He is a well-known figure in the cricketing world. During

1958-59 he was manager of the M.C.C. team touring Australia and New Zealand.

★ **Mr. H. A. Ford** has become manager of nitrogen products of the chemical division of Olin Mathieson Chemical Corporation. He will be responsible for co-ordinating all nitrogen products marketing of the division.

★ **Mr. E. W. Molesworth** has been appointed chief engineer for the London, southern and eastern areas of G.E.C. Ltd. His predecessor in this position, **Mr. C. J. O. Garrard**, was recently appointed assistant general manager of the company's Erith engineering works.



E. F. D. Webb.



H. McGlyn.

★ **Mr. E. N. Griffith** has been elected vice-president of the British Engineers' Association after having served on its governing council for the past 10 years as the representative of the Agricultural Engineers Association, of which association he was president from 1948 to 1954. He is the chairman and joint managing director of Rotary Hoes Ltd., and is a governor of the National College of Agricultural Engineering.

★ Organisational changes have taken place in the elastomer chemicals department of the Du Pont Co. (U.K.) Ltd. **Mr. W. P. Fletcher**, formerly manager of the elastomer research laboratory at Hemel Hempstead, has been appointed assistant elastomers sales manager, **Mr. D. J. B. Coulter**, formerly technical sales representative in the Manchester area, has been appointed in his place and **Dr. J. Mausner**, formerly of the Hemel Hempstead laboratory, has been appointed sales representative in the Manchester area.

★ **Mr. E. F. D. Webb** has been appointed engineering manager of Hagan Controls Ltd. He took up his appointment on his recent return

from America where he has been studying American methods of automatic process control.

★ **Mr. J. G. Thorpe** has been appointed sales manager of Semi-conductors Ltd. He has worked for the Armament Research Establishment and the Royal Radar Establishment at Malvern as a senior scientific officer engaged on basic circuit techniques.

★ **Prof. C. J. Bakker**, the director general of C.E.R.N., died in an aeroplane accident in April. He studied physics under Zeeman in Amsterdam. He was one of the eight experts who in 1951 drew up the plans for the future C.E.R.N. and when the interim organisation was started in 1952 he was appointed director of the synchro-cyclotron group. At the first session of the council of the new organisation he became a member of the directorate and a director of the synchro-cyclotron division on a part-time basis. In 1955 he was appointed full-time deputy director general of C.E.R.N. and later that year he became director general of the organisation in succession to Prof. Bloch.

★ **Mr. G. F. Ashford** has been appointed deputy chairman of the chemical group of the Distillers Co. Ltd. **Mr. J. M. Rimington**, managing director of the chemical division, has retired for reasons of health after 30 years' service, and will be succeeded by **Mr. J. H. Dunn**, who will be succeeded by **Mr. J. S. Hunter** as assistant managing director (commercial) of the chemical division of the company.



J. F. Willsher.



S. L. Waide.

★ **Mr. D. M. Boyd**, who has been a director of Fisons Ltd. for the past 18 years, has retired from the board. He joined Doughty Richardson Fertilizers Ltd., one of the constituent companies of the group in 1919, and became a director of that company in 1933. He will be remaining with the company for a further year to complete his work with the Association of Chemical and Allied Employers, of which he is chairman.

World News

AUSTRALIA

High-quality lubricants

The British Petroleum Co. Ltd. is in an advanced stage of planning a project for the construction of a lubricants oil refinery. The project envisages production of up to 100,000 tons p.a. of high-quality lubricants. Although figures have not been finalised it is expected that the plant will cost between £A8 and 10 million.

DENMARK

Polyolefine manufacture

A joint company is to be formed in Denmark for the manufacture of polyolefines. Initially, *Alkathene*, the brand of polyethylene developed by I.C.I., will be made, using their high-pressure process.

Partners in this new enterprise are I.C.I. Ltd. and A/S Dampskibsselskabet Svendborg and Dampskibsselskabet af 1912 A/S, owners of the Maersk refinery, Copenhagen.

The new company, to be known as Danbritkem A/S, Copenhagen, will have a capital of about 80 million Danish kroner (approximately £4 million sterling), provided equally by the Danish and British partners. The polyethylene plant, to be built alongside the Maersk refinery, will have a capacity of 15,000 tons p.a.

BELGIUM

Phthalic anhydride

A contract for the design, engineering and construction of a 5,000-metric-ton/yr. phthalic anhydride plant has been awarded to Badger N.V., The Hague, by Union Chimique Belge S.A., Brussels. Badger N.V. is a wholly owned subsidiary of the American Badger Manufacturing Co.

The new phthalic plant will be constructed at Schoonarde at an estimated cost of \$2 million.

NEW ZEALAND

Ironsand export

The Japanese steel industry is showing interest in imports of New Zealand ironsand. Mr. Ochikawa, managing director of the Niisho Seiko Kaisha, a Japanese steel mill, mentioned that Japan would like to buy ironsand from New Zealand if the New Zealand Government removed the present export ban and export prices were reasonable. There are deposits of several hundred million tons of good-

quality ironsand in the coastal area near New Plymouth, in the North Island. The ironsand could be used for steel production without dressing or concentrating, he added. There are no ironsand imports into Japan.

Aluminium smelting project

Discussions between the representatives of Consolidated Zinc Pty., which is expected to build the £100-million aluminium smelting scheme in Southland, and senior Ministry of Works officials took place at Invercargill.

The conference followed a series of meetings between officers of the two parties in recent weeks. The talks are to make an up-to-date assessment of investigations already carried out so that the company can more easily determine the cost factors involved, particularly regarding the development of the required amount of electric power from Lake Manapouri.

FRANCE

Hydrogen plant

A plant for the production of hydrogen gas is to be built near Lyon for Rhodiaceta, a subsidiary of Rhone-Poulenc. The plant will produce 3,000 cu.m./hr. of hydrogen gas by reforming Lacq natural gas. The resultant gas produced will be extremely pure, containing less than 5 p.p.m. of carbon monoxide. Chemical Construction (G.B.) Ltd. will provide the design, engineering and construction of the plant which is scheduled to start operating early next year.

DOMINICAN REPUBLIC

Acetylene plant

A new acetylene plant has been opened in San Cristobal. The plant cost £30,000 to equip and will have a production rate of 800 cu.ft./hr.; 500 cylinders will be held in stock for the use of the armed forces and private industry.

INDIA

Yeast factory

The Distillers Co. Ltd. (D.C.L.) and Shaw Wallace & Co. Ltd. of Calcutta have formed the India Yeast Co. (Private) Ltd., in which D.C.L. will have a minority interest. The company is building a factory at Calcutta and will produce dried and compressed baker's yeast, using molasses as the main nutrient material.

D.C.L. has been producing yeast in the U.K. since about 1886 and will provide technical knowledge for the design and erection of this plant and for its operation, initially from the strain of pure yeast grown by the D.C.L. research laboratories. It is expected that the factory will be in production by early next year.

NORWAY

Leakage in reactor

The heavy water reactor at the Norwegian Institute of Atomic Energy at Kjeller has been stopped after a leakage had been registered. The uranium rods will now be removed and 'cleaned' in the Institute's new purification establishment. An estimate of how long the repairs will take cannot be arrived at until this has been done, and it has been found out where the heavy water is leaking out. The reactor, JEEP I, has been in continual use for 10 years. Work on a new reactor, JEEP II, has just been started and is expected to be completed in three years' time.

ARGENTINA

Phenol plant planned

A new, jointly owned, phenol plant is being planned by Hooker Chemical Corporation and Atnor, Compania Nacional para la Industria Quimica S.A.M. of Argentina. A new company, Duranor, was formed for this purpose in January.

The phenol facility is to be built at one of Atnor's two established plant locations. Construction is expected to begin in July and completion is planned for less than two years.

UNITED STATES

Melamine expansion

Allied Chemical has doubled facilities at Toledo, U.S.A., for making melamine and urea moulding compounds. It is not expected that full-scale production of 45 million lb. will be reached until the late 1960s.

The combined expansion is based on the complex nature of melamine and urea production, which means that expansion, once started, must be carried out all along the line. The company reports that more than 90% of the output of all melamine resin is now used in the plastic dinnerware industry. The remainder is sold mainly for electrical fixtures and wiring devices.

Melamine dinnerware, made by 30 manufacturers in the U.S.A. and Canada, constitutes nearly one-quarter of all dinnerware sold today and totals \$85 million on a retail level.

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